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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held on Wednesday, December 5th, at the Chemical Society's Rooms, Burlington House. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Frank Knowles, Archibald Knox, A.I.C., Charles Roger Middleton, B.Sc., A.R.C.Sc., D.I.C., A.I.C., Harold Richard Read, A.I.C , George Hogan, F.I.C., and Thomas Francis Doyle.

Certificates were read for the second time in favour of:—Messrs. Robert Charles Frederick and Hubert Thomas Stanley Britton, M.Sc. (Lond.), F.I.C.

The following were elected Members of the Society:—Messrs. Laurence Eversley Campbell, M.Sc. (Lond.), F.I.C., John Troubridge Hannen, B.A. (Cantab), A.R.C.Sc., A.I.C., Cyril Langley Hinton, F.I.C., Douglas William Kent-Jones, B.Sc. (Lond.), F.I.C., Thomas William Alan Shaw, M.Sc. (Liv.), William Hall Simmons, A.I.C., Kenneth Edward Nethercoate Williams, and Percy Noel Williams, M.Sc. (Liv.), A.I.C.

The following papers were read:—"Crystalline Bromides of Linseed Oil," by Harold Toms, M.Sc. (under the Analytical Investigation Scheme); "The Plea for Standardisation," by M. S. Salamon, B.Sc.; "Note on the Estimation of Chromium," by Hubert T. S. Britton, M.Sc., F.I.C.; and "The Colorimetric Estimation of Lead in Cream of Tartar," by R. L. Andrew.

The Application of the Iodimetric Method to the Analysis of Sugar Products.

By C. L. HINTON, F.I.C., and T. MACARA, F.I.C.

(*Read at the Meeting, October* 3, 1923.)

During the last few years the iodimetric process for the estimation of reducing sugars has been examined by a number of investigators, the chief of these being Willstätter and Schüdel (*Ber.*, 1918, **51**, 780; Analyst, 1918, **43**, 416); H. M. Judd (*Biochem. J.*, 1920, **14**, 255; Analyst, 1920, **45**, 224); Baker and Hulton (*Biochem. J.*, 1920, **14**, 754); and Cajori (*J. Biol. Chem.*, 1922, **54**, 617 ; Analyst, 1923, **48**, 73). (See also note at end.)

The process consists essentially in adding a known amount of standard iodine solution to a given quantity of the dilute sugar solution, rendering the mixture alkaline and allowing it to stand for a short time, then re-acidifying and titrating the excess of iodine with standard thiosulphate solution. In most cases, sodium hydroxide is used for making the solution alkaline, though Cajori prefers sodium carbonate, as being less likely itself to affect the sugars. As will be seen, quite definite results can be obtained with sodium hydroxide, and this has been used in the present experiments. A few experiments on the effect of other alkalis are, however, described at the end of this paper. The alkali should always be added after the mixing of the sugar and iodine solutions.

The whole series of operations is carried through quite rapidly, and with a minimum of apparatus and reagents which are readily available in any laboratory, so that the process recommends itself admirably for factory analysis. Unfortunately the statements as to the extent to which the different sugars are oxidised by the alkaline iodine solution are not altogether in agreement, and it was as an attempt to clear up these points and to place the iodimetric analysis of sugar mixtures on a sound basis, that the present work was carried out.

General Experimental Procedure.—The required amount of sugar solution was measured into a 250 c.c. conical flask, and made up to approximately 50 c.c. with distilled water; 40 c.c. of 0·05 *N* iodine solution were then added, followed by the required amount of 0·5 *N* sodium hydroxide solution, and the flask stoppered. After standing for the time required, the mixture was acidified with 5 c.c. of 2 *N* sulphuric acid and immediately titrated with 0·05 *N* thiosulphate solution, accurately standardised against pure potassium iodate. Starch was used for the end-point. In all cases one or more blank experiments, in which distilled water was used in place of the sugar solution, were carried out at the same time and under the same conditions as each series of experiments.

I. DEXTROSE.

Most observers find a quantitative oxidation of dextrose to gluconic acid by alkaline iodine, according to the equation:

$$C_6H_{12}O_6 + I_2 + 3NaOH = C_5H_{11}O_5COONa + 2NaI + 2H_2O.$$

1 grm. of dextrose should therefore require 1·410 grm. of iodine for oxidation.

Miss H. M. Judd, however, reports finding only 1·315 grm. iodine required per 1 grm. of dextrose, and concludes that the oxidation of dextrose is never complete, though a definite and constant weight of iodine always reacts with a given weight of dextrose.

A. PRELIMINARY EXPERIMENTS.—Some preliminary work, to fix the experimental conditions, was carried out on a sample of nearly pure commercial dextrose. This material gave the following analytical figures:—Moisture, 2·52 per cent.; $[\alpha]_D$ (15 per cent. concn.) +51·0°; $[\alpha]_D$ on dry basis = +52·3°.

(*a*) *Variation in Amount of Alkali Used.*—A 0·2 per cent. solution of the dextrose was prepared, and quantities of 40 c.c., made up to 50 c.c. before adding the iodine, were used for each titration. Varying quantities of 0·5 *N* soda were then added, the flasks stoppered, and the solutions allowed to stand at room temperature for 10 minutes; 5 c.c. of 2 *N* sulphuric acid were then added, and the solutions at once titrated.

The results are collected in Table I.

TABLE I.—EFFECT OF VARIATION IN AMOUNT OF ALKALI USED.

Amount of sugar in each titration:—0·08 grm.
Time of reaction:—10 minutes.
Blank titration:—39·13 c.c. of 0·05 *N* thiosulphate solution.

No.	Sodium hydroxide (0·5 *N*) solution. c.c.	Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	3	25·12	14·01	1·112
2	4	22·14	16·99	1·348
3	5	21·96	17·17	1·362
4	6	22·02	17·11	1·357
5	7	22·14	16·99	1·348
6	9	22·72	16·41	1·302
7	10	23·13	16·00	1·270

In No. 1 the amount of sodium hydroxide is obviously deficient. No. 3, where 5 c.c. of 0·5 *N* sodium hydroxide solution were used, gives the maximum iodine value for the sugar. In this case the ratio of iodine to sodium hydroxide in terms of normal concentrations is 1:1·25, and this amount of sodium hydroxide approximately corresponds to the amount necessary for the neutralisation of the gluconic and hydriodic acids formed by oxidation of the sugar, with sufficient excess for the formation of hypoiodite with all the excess of iodine:

$$I_2 + 2NaOH = NaOI + NaI + H_2O.$$

Alkali in excess of this amount diminishes the apparent iodine value of the dextrose, just as does a deficiency. Even a ratio of 1:1·5, as shown by No. 4, shows a slightly low value, although this is the proportion usually recommended. This would theoretically be the amount of sodium hydroxide required if the whole of the iodine entered into the glucose reaction (given by first equation above), but as in practice at least half of the iodine is unattacked, a certain slight excess of alkali must be present if the ratio 1:1·5 is maintained.

(*b*) *Proportions of Dextrose to Iodine.*—In this series varying amounts of dextrose solution were measured out, made up to 50 c.c., and the iodine and sodium hydroxide added. The time of reaction was 10 minutes. For Nos. 1 to 5, 6 c.c. of sodium hydroxide solution were used; for the others, 5 c.c.

The results, given in Table II., show that, provided not more than about half of the total iodine is used up, the proportion of dextrose to iodine does not affect the result. Obviously, it is an advantage to work with as large a quantity of sugar as possible, whilst remaining within safe limits. A suitable amount would be 0·08 grm. dextrose for 40 c.c. of 0·05 *N* iodine.

TABLE II.—EFFECT OF VARYING EXCESS OF IODINE USED.

Time of reaction:—10 minutes.
Blank titration:—39·13 c.c. 0·05 *N* thiosulphate solution.

No.	Dextrose. Grm.	Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
6 c.c. of 0·5 *N* NaOH used.				
1	0·02	34·86	4·27	1·355
2	0·04	30·55	8·58	1·361
3	0·05	28·42	10·71	1·359
4	0·08	22·07	17·06	1·353
5	0·08	22·02	17·11	1·357
5 c.c. of 0·5 *N* NaOH used.				
6	0·08	21·96	17·17	1·362
7	0·10	17·78	21·35	1·355
8	0·12	13·95	25·18	1·332

(*c*) *Time of Reaction.*—Willstätter and Schüdel recommend that 12 to 20 minutes be allowed for the reaction to take place. Baker and Hulton state that 3 to 5 minutes suffice. From the experiments detailed in Table III., in which varying periods were used, it would appear that 5 minutes may be insufficient in the case of dextrose. There appears, however, to be no appreciable action after 10 minutes.

TABLE III.—EFFECT OF TIME OF REACTION.

Amount of sugar in each titration:—0·08 grm.
Blank titration:—39·13 c.c. of 0·05 *N* thiosulphate solution.

No.	Time.	Thiosulphate (0·05 *N*) solution. required. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
6 c.c. of 0·5 *N* NaOH.				
1	5 mins.	22·57	16·56	1·314
2	10 „	22·02	17·11	1·357
3	20 „	22·05	17·08	1·355
5 c.c. of 0·5 *N* NaOH.				
4	10 „	21·96	17·17	1·362
5	20 „	21·94	17·19	1·364

(*d*) *Concentration of Reacting Mixture.*—For this purpose a 0·8 per cent. solution of the dextrose was prepared, and in each case 10 c.c. were accurately measured out and made up with distilled water to the required volume. The results in Table IV. show slightly higher figures for Nos. 1, 2 and 5. This is probably due to slight loss of iodine by volatilisation, from these more concentrated mixtures. The total volume of reacting solutions should conveniently be about 100 c.c. per 40 c.c. of 0·05 *N* iodine used. Nos. 5, 6 and 7 are the results of an attempt to ascertain whether increasing the dilution of the solution had any effect on the influence of an excess of alkali in diminishing the iodine value; this does not appear to be the case.

TABLE IV.—EFFECT OF CONCENTRATION OF SOLUTION.

Amount of sugar in each titration:—0·08 grm.
Blank titration:—38·58 c.c.
Time of reaction:—10 minutes.

No.	Vol. of sugar solution. c.c.	Total vol. c.c.	Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
5 c.c. of 0·5 *N* NaOH.					
1	10	55	21·35	17·23	1·367
2	30	75	21·38	17·20	1·365
3	50	95	21·41	17·17	1·362
4	70	115	21·41	17·17	1·362
8 c.c. of 0·5 *N* NaOH.					
5	30	78	22·10	16·48	1·308
6	50	98	22·14	16·44	1·304
7	70	118	22·13	16·45	1·305

B. **Iodine Reducing Power of Pure Dextrose.**—The commercially pure dextrose used above was recrystallised from dilute alcohol, the first crop of crystals being rejected and only the middle crystallising portion of the sample being retained. This was washed on a Büchner funnel with 70 per cent. alcohol, absolute alcohol and ether, and dried initially at 45° C., with gradual raising of the temperature to 95° C. About 10 per cent. of the original dextrose was thus recovered, which gave the following figures on analysis:—Moisture (105° C.), 0·21 per cent.; ash, 0·07 per cent.; $[\alpha]_D^{20}$ const. (5 per cent. solution) $+52{\cdot}67°$ C.; on dry basis $= +52{\cdot}78°$ C.)

For the determination of the iodine value a 0·8 per cent. solution of this dextrose was prepared, and 10 c.c. accurately measured out and made up to 50 c.c. before adding the 40 c.c. iodine solution; 5 c.c. of 0·5 *N* sodium hydroxide solution were used, and the time of reaction was 10 minutes. The results are shown in Table V.; Nos. 2, 3 and 4 were carried out on a different solution and on a different occasion from No. 1. It will be seen that the average iodine reduction for this sample is 1·406 grms. per 1 grm. of sugar, which corresponds to a value of 1·410 grms. for dry ash-free dextrose. This is the theoretical value. The accuracy of a single determination appears to be about ±0·2 per cent.

TABLE V.—IODINE REDUCING POWER OF DEXTROSE.

Amount of sugar in each titration:—0·08 grm.
Amount of alkali in each titration:—5 c.c. of 0·5 *N* NaOH.
Time of reaction:—10 minutes.

No.	Blank titration. Thiosulphate (0·05 *N*) solution. c.c.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	39·50	21·79	17·71	1·405
2	39·21	21·45	17·76	1·409
3	,,	21·51	17·70	1·404
4	,,	21·47	17·74	1·407
			Average	1·406
			on dry ash-free basis	1·410

In order to verify the experiments already carried out on the commercial dextrose as to the influence of varying excess of iodine, a series of titrations was carried out with the use of varying proportions of dextrose to iodine (Table VI.). Nos. 1 and 2 were made in the usual way outlined above; the others were done by measuring out arbitrary quantities of dextrose solution, making the liquids up to 100 c.c. and using 40 c.c. of 0·1 *N* iodine solution and 10 c.c. of 0·5 *N* sodium hydroxide solution.

TABLE VI.—EFFECT OF VARYING EXCESS OF IODINE.

Nos. 1 and 2 carried out with 40 c.c. of 0·05 *N* iodine + 5 c.c. of 0·5 *N* NaOH.
Others with 40 c.c. of 0·1 *N* iodine + 10 c.c. of 0·5 *N* NaOH.
Time of reaction:—10 minutes.

No.	Dextrose. Grms.	Blank. Thiosulphate (0·05 *N*) solution. c.c.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	0·0500	39·38	28·31	11·07	1·405
2	0·0500	,,	28·30	11·08	1·406
3	0·05087	82·00	70·74	11·26	1·405
4	0·0846	,,	63·21	18·79	1·409
5	0·1245	,,	54·43	27·57	1·405
6	0·1472	,,	49·31	32·69	1·409
				Average	1·406(5)

Within the range of proportions used, there is no appreciable difference whether a smaller or larger amount of dextrose be taken. It must be noted, however, that in all these cases there is an excess of iodine greater than the total amount reduced. The average figure practically agrees with that of the previous series, with about the same degree of accuracy.

In view of the ease with which dextrose is quantitatively oxidised by alkaline iodine, it is difficult to account for Miss H. M. Judd's statement that the reaction is never complete.

II. LACTOSE.

A sample of pure milk sugar (lactose hydrate) was used, with the following analysis:—Moisture, 0·025 per cent.; ash, 0·065 per cent.; $[\alpha]_D^{20}$ (c = 5) + 52·3° C.

(*a*) *Variation in Amount of Alkali.*—A 0·4 per cent. solution was prepared, 40 c.c. being used for each titration, and made up to 50 c.c. before the iodine was added. Amounts of 0·5 *N* sodium hydroxide solution varying from 3 to 8 c.c. were used; the reaction time was 10 minutes. Table VII. shows that the maximum iodine consumption occurs when the ratio of iodine to sodium hydroxide is 1:1·13 to 1:1·25. In these cases the alkali is approximately the amount required for the neutralisation of the sugar acid and hydriodic acid found, and formation of hypoiodite with the excess of iodine, as was found in the case of dextrose.

TABLE VII.—LACTOSE. EFFECT OF VARYING PROPORTIONS OF ALKALI.

Amount of sugar in each titration:—0·16 grm.
Time of reaction:—10 minutes.

No.	Sodium hydroxide (0·05 *N*) solution. c.c.	Blank. Thiosulphate (0·05 *N*) solution. c.c.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	3	39·05	26·13	12·92	0·512
2	4	39·09	22·00	17·09	0·678
3	4·5	39·09	21·31	17·78	0·705
4	5	39·05	21·37	17·68	0·701
5	6	39·05	21·77	17·28	0·685
6	7	39·09	22·33	16·76	0·665
7	8	39·05	23·02	16·03	0·635

(*b*) *Proportions of Lactose to Iodine.*—Table VIII. shows the results for lactose when the proportion of sugar is varied, the iodine and alkali being kept constant. As with dextrose, there is no appreciable effect due to the amount of sugar, provided there is an excess of iodine at least equal to the amount reduced. In No. 5 the amount of iodine is obviously deficient. A convenient amount of lactose to use would appear to be 0·16 grm. per 40 c.c. of 0·05 *N* iodine.

TABLE VIII.—LACTOSE. EFFECT OF VARYING EXCESS OF IODINE.

Amount of alkali used:—5 c.c. of 0·5 *N* NaOH.
Time of reaction:—10 minutes.
Blank titration:—39·05 c.c. of 0·05 *N* thiosulphate solution.

No.	Sugar. Grms.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	0·04	34·63	4·42	0·701
2	0·08	30·16	8·89	0·704
3	0·10	27·98	11·07	0·703
4	0·16	21·39	17·66	0·701
5	0·20	17·69	21·36	0·678

(*c*) *Time of Reaction.*—The few experiments recorded in Table IX. show that with lactose, as with dextrose, 5 minutes appears to be rather insufficient for the completion of the reaction. On the other hand, No. 3 points to the possibility of a reduction of iodine beyond the theoretical value, if the time is prolonged; but the present experiments are perhaps inadequate to decide this point.

Lactose hydrate should have an iodine reducing power of 0·705 grm. of iodine per grm., corresponding to 0·743 grm. for anhydrous lactose. Miss H. M. Judd's figure of 1·502, and even that of Baker and Hulton (0·762), for anhydrous lactose, are higher than the figures found in these experiments, which show no reason for supposing that the theoretical figure does not hold good for the conditions arrived at here.

TABLE IX.—LACTOSE. EFFECT OF TIME OF REACTION.

Amount of sugar in each titration:—0·16 grm.
Amount of alkali:—5 c.c. of 0·5 N sodium hydroxide solution.
Blank titration:—39·09 c.c. of 0·05 N thiosulphate solution.

No.	Time.	Back titration. Thiosulphate (0·05 N) solution. c.c.	Iodine (0·05 N) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	5 min.	21·57	17·52	0·695
2	10 ,,	21·41	17·68	0·701
3	20 ,,	21·22	17·87	0·709

III. SUCROSE.

There is some disagreement in the statements as to the behaviour of sucrose with alkaline iodine. Willstätter and Schüdel found no oxidation, whilst Miss Judd found 0·006 grm. of iodine reduced per 1 grm. of sucrose, and Baker and Hulton found 0·02 grm. per grm. of sucrose; both these figures were obtained, however, on amounts of 0·1 grm. of sucrose, and therefore with very small amounts of iodine taken up.

The sucrose used in the following experiments was a sample of pure sucrose supplied by Messrs. Tate for standardisation purposes. It contained 0·01 per cent. of moisture and 0·008 per cent. of ash, and polarised + 66·55° at 16° C., and at a concentration of 20 per cent.

(*a*) *Effect of Varying Proportions of Alkali.*—The sucrose was finely powdered and weighed out separately for each titration; it was dissolved in 50 c.c. of water, and the iodine and alkali added as usual. The time was kept at 10 minutes. As will be seen from Table X., the amount of alkali appeared to have little influence, even when as much as 20 grms. of sucrose was used.

TABLE X.—SUCROSE. EFFECT OF VARYING PROPORTIONS OF ALKALI.

Amount of sugar in each case:—20 grms.
Time of reaction:—10 minutes.
Blank titration:—38·90 c.c. of 0·05 N thiosulphate solution.

No.	0·5 N NaOH c.c.	Back titration. Thiosulphate (0·05 N) solution. c.c.	Iodine (0·05 N) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grms.
1	3	31·96	6·94	0·0022
2	5	31·32	7·58	0·0024
3	8	30·78	8·12	0·0026

(*b*) *Effect of Varying Proportions of Sucrose to Iodine.*—Experiments in which different amounts of sucrose for the same amount of iodine were used showed that the iodine reduction is not strictly in proportion to the sucrose used. There is a slight falling off with increasing amounts of sucrose, as Table XI. shows. This at once precludes the possibility of the reduction being due to traces of dextrose; for, if this were so, the reduction should be practically proportional to the sugar used.

TABLE XI.—SUCROSE. EFFECT OF VARYING PROPORTIONS OF SUCROSE TO IODINE (1).

Amount of alkali used:—6·5 c.c. of 0·5 *N* NaOH.
Time of reaction:—10 minutes.
Blank titration:—39·06 c.c. of 0·05 *N* thiosulphate solution.

No.	Sucrose. Grms.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sucrose. Grm.
1	2	37·83	1·23	0·0038
2	5	36·40	2·66	0·0034
3	10	34·18	4·88	0·0031
4	15	32·30	6·76	0·0029
5	20	30·42	8·64	0·0027

It appeared probable that the lower iodine reduction with the larger amounts of sucrose might be connected with the smaller excess of iodine. In order to examine this point, additions of 0·08 grm. of the dextrose used in Tables I. to IV. were made to two quantities of 4 and 8 grms. of sucrose. To allow sufficient iodine 50 c.c. of 0·05 *N* iodine solution were used in these cases, with 6·25 c.c. of 0·5 *N* sodium hydroxide solution. From the amounts of iodine reduced were deducted the amounts due to the dextrose, and the difference gave the iodine value of the sucrose used. (Table XII.) As anticipated, the figures were rather lower than when no dextrose was used, though not so low as might have been expected if the whole of the used-up iodine had been reduced by sucrose.

TABLE XII.—SUCROSE. EFFECT OF VARYING PROPORTION OF SUCROSE TO IODINE (2).

0·08 grm. commercially pure dextrose (Table I.) present in each case.
Iodine equiv.:—17·17 c.c. of 0·05 *N* iodine solution.
Amount of alkali used:—6·25 c.c. of 0·5 *N* NaOH.
Time 10 minutes. Blank titration, 49·38 c.c.

No.	Sucrose. Grms.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Less amount due to dextrose. c.c.	Iodine per 1 grm. of sucrose. Grm.
1	4	30·34	19·04	1·87	0·0030
2	8	28·83	20·55	3·38	0·0027

(*c*) *Effect of Temperature.*—Certain irregularities in the figures so far obtained for sucrose (*e.g.* compare Table X. with No. 4 of Table XI.) led to an investigation into the effect of temperature on the reaction, since it might be expected that here, dealing with a very incomplete oxidation, the temperature might considerably influence the extent to which the oxidation proceeded.

For this series the sucrose was dissolved in water and the temperature adjusted by means of a bath; the iodine and alkali were then added (at room temperature) and the mixture agitated in the bath for 3 minutes and allowed to stand in the bath for the remaining 7 minutes.

TABLE XIII.—SUCROSE. EFFECT OF TEMPERATURE.

Amount of sugar in each case:—20 grms.
Amount of alkali:—5 c.c. of 0·5 *N* NaOH.
Blank titration:—39·91 c.c. of 0·05 *N* thiosulphate solution.
Time:—10 minutes.

No.	Temperature. ° C.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grm.
1	15	30·73	9·18	0·0029
2	22·5	25·42	14·49	0·0046
3	30	21·40	18·51	0·0059

The results (Table XIII.) showed a considerable increase of iodine reduction due to increased temperature; the iodine value at 30° C. was double that at 15° C., with an intermediate value at 22·5° C.

Accordingly, for further experiments on sucrose a standard temperature of 17·5° C. (the most suitable at the time this work was carried out) was adopted.

(*d*) *Effect of Time of Reaction.*—For the reason given in the preceding section, it might be expected that the reaction of sucrose with alkaline iodine would proceed much further when the time was prolonged. The point was investigated by allowing quantities of 10 grms. to react for increasing periods of 10 minutes, ½ hour, and 2 hours, with a blank in each case standing the same length of time. There was a rapid increase of reduction in the first half-hour, the rate of which, however, fell away considerably in the succeeding 1½ hours. A fourth mixture was then prepared and allowed to stand for 3 days at 17·5° C. There was no further increase, although the excess of iodine was considerable. The four experiments are recorded in Table XIV.; it appears that a maximum value for the iodine reduction is reached after about 2 hours, to the extent of 0·0067 grm. of iodine per 1 grm. of sugar.

TABLE XIV.—SUCROSE. EFFECT OF TIME OF REACTION.

Amount of sugar in each case:—10 grms.
Amount of alkali:—5 c.c. of 0·5 *N* NaOH.
Temperature:—17·5° C.

No.	Time.	Blank. Thiosulphate (0·05 *N*) solution. c.c.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grm.
1	10 mins.	40·16	35·17	4·99	0·0032
2	30 mins.	40·14	30·90	9·24	0·0059
3	2 hrs.	40·12	29·48	10·64	0·0067
4	3 days	40·14	29·63	10·51	0·0067

(*e*) *Iodine Reducing Power of Sucrose.*—From the data given in Tables X. to XIV., it is possible to obtain a value for the reducing power of sucrose under the conditions of the standard procedure. This procedure, as originally outlined, must now be modified in cases where sucrose is present, by the adoption of a standard temperature, say 17·5° C. At this temperature, the iodine reduction of 10 grms. of sucrose for 10 minutes in presence of 40 c.c. of 0·05 N iodine solution and 5 c.c. of 0·5 N sodium hydroxide solution is 0·0032 grm. per 1 grm. of sucrose. The figures of Tables X., XI. and XII. are rather lower than this; but these were obtained at room temperature which, although not taken at the time, was certainly lower than 17·5° C.

For practical purposes it will be a sufficiently close approximation to take the figure 0·003 for the iodine value of sucrose. The figure obviously only becomes of importance at all in analysis when the amount of sucrose is relatively large compared with those of the sugars which are completely oxidised.

IV. LÆVULOSE.

Most observers have reported no oxidation of lævulose by alkaline iodine, but Miss Judd and Baker and Hulton found values of 0·10 grm. of iodine per grm. of lævulose. If this figure is correct, it will obviously seriously affect any attempts at estimation of dextrose in presence of lævulose.

Miss Judd gives no data as to the lævulose used, merely stating that its purity was tested by the polarimeter. The sample used by Baker and Hulton is described as having been recrystallised from alcohol, having $[\alpha]_D = -88{\cdot}4$ and a reducing power with Fehling solution, calculated as lævulose, of 99 per cent. Inasmuch as the $[\alpha]_D^{20}$ of lævulose at a concentration of 10 per cent. is $-92{\cdot}9°$ C. (Vosburgh), it appears doubtful whether their sample could have been entirely free from dextrose.

For these experiments a sample of lævulose purchased as pure, and having $[\alpha]_D^{20}$ $-85{\cdot}6°$ C. on dry basis, in 20 per cent. solution, was recrystallised by the method described by T. S. Harding (*J. Amer. Chem. Soc.*, 1922, **44**, 1755). The product from the second crystallisation had the following analysis:—Moisture, 0·4 per cent.; ash, 0·02 per cent.; $[\alpha]_D^{20}$ (20 per cent. solution), $-94{\cdot}1°$ C. (on dry basis). (Vosburgh's formula for this temperature and concentration requires $-94{\cdot}3°$ C.)

(*a*) *Effect of Varying Proportions of Alkali.*—The lævulose in these experiments was separately weighed out for each titration, and dissolved in 50 c.c. of water. From the results of Table XV. it appears that an excess of alkali causes a considerably increased oxidation of lævulose. This is to be expected in view of the readiness with which lævulose is attacked by alkalis. A few experiments were also carried out in which a small amount of dextrose was used in addition to the lævulose; in this case 50 c.c. of 0·05 *N* iodine solution were used (see Table XVI.). From the amounts of iodine reduced were deducted the amounts required by the dextrose for the respective proportions of alkali used; the remainder was taken as being the iodine reduction of the lævulose. As before, excess of alkali caused an increase in the oxidation of the lævulose.

TABLE XV.—LÆVULOSE. EFFECT OF VARYING PROPORTIONS OF ALKALI (1).

Amount of lævulose in each case:—2 grms.
Time of reaction:—10 minutes.
Blank titration:—40·32 c.c. of 0·05 *N* thiosulphate solution.

No.	NaOH (0·5 *N*) solution. c.c.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugar. Grm.
1	4	38·27	2·05	0·0065
2	5	37·80	2·52	0·0070
3	8	36·43	3·89	0·0124

TABLE XVI.—LÆVULOSE. EFFECT OF VARYING PROPORTIONS OF ALKALI (2).

Amount of lævulose in each case:—5 grms.
0·08 grm. of the commercially pure dextrose (Table I.) also present.
Time:—10 minutes.
Blank titration:—49·95 c.c. of 0·05 *N* thiosulphate solution.

No.	NaOH (0·5 *N*) solution. c.c.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Less amount due to dextrose (Table I.). c.c.	Iodine per 1 grm. of lævulose. Grm.
1	5	30·41	19·54	2·55	0·0032
2	6·25	29·17	20·78	3·61	0·0046
3	8	28·48	21·47	4·41	0·0056

(*b*) *Effect of Temperature.*—Before proceeding further with the experiments on lævulose, the temperature effect was examined, as this also might be considerable, as with sucrose. Accordingly 1 grm. quantities of lævulose were treated by the standard method at temperatures of from 14·5° to 25° C., the same procedure being adopted as for sucrose. The temperature effect was even greater than for sucrose, the iodine value being trebled between these extremes (see Table XVII.). It was evidently necessary here also to fix a standard temperature, and 17·5° C. was therefore used for the further experiments.

TABLE XVII.—LÆVULOSE. EFFECT OF TEMPERATURE.

Amount of lævulose in each case:—1 grm.
Amount of alkali:—5 c.c. of 0·5 N NaOH.
Time:—10 minutes.
Blank titration:—39·31 c.c. of 0·05 N thiosulphate solution.

No.	Temp. ° C.	Back titration. Thiosulphate (0·05 N) solution. c.c.	Iodine (0·05 N) solution reduced. c.c.	Iodine per 1 grm. of lævulose. Grm.
1	14·5	38·23	1·08	0·0068
2	17·5	37·59	1·72	0·0109
3	19	37·26	2·05	0·0130
4	25	36·08	3·23	0·0205

(*c*) *Effect of Varying Proportions of Lævulose to Iodine.*—As with sucrose, the experiments designed to show the effect of increasing quantities of lævulose in proportion to iodine, showed a falling off in the iodine reduction as the amount of sugar was increased (Table XVIII.). (No. 4 of this series is included, although the temperature was not noted, as it was determined on a separate small quantity of lævulose which had been specially recrystallised from the original material. Its polarisation on the dry basis agreed with that required by Vosburgh's formula. The temperature was probably rather on the low side.) This decrease is most marked for smaller amounts of sugar, and there appears to be a tendency to approximate to a constant figure of about 0·0065.

TABLE XVIII.—LÆVULOSE. EFFECT OF VARYING PROPORTIONS OF LÆVULOSE TO IODINE (1).

Amount of alkali used:—5 c.c. of 0·5 N NaOH.
Time:—10 minutes.
Temp.:—17·5° C. (except in 4).
Blank titration:—39·31 c.c. of 0·05 N thiosulphate solution.

No.	Lævulose. Grms.	Back titration. Thiosulphate (0·05 N) solution. c.c.	Iodine (0·05 N) solution reduced. c.c.	Iodine per 1 grm. of lævulose. Grm.
1	0·16	38·86	0·45	0·018
2	1·0	37·59	1·72	0·0109
3	2·0	36·57	2·74	0·0087
4	2·58	37·22 (Blank 40·16)	2·94	0·0072
5	5·0	34·03	5·28	0·0067

If dextrose is also present in amount sufficient to reduce an appreciable quantity of the iodine, the oxidation of the lævulose is diminished. Table XIX. shows the results found for various quantities of lævulose when 0·06 grm. of dextrose was also present. Here again the falling off in reducing power becomes less marked as the amount of lævulose increases, and appears to tend to a constant value of about 0·004.

TABLE XIX.—LÆVULOSE. EFFECT OF VARYING PROPORTIONS OF LÆVULOSE TO IODINE (2).

Amount of pure dextrose (Table V.) present in each case:—0·06 grm.
Amount of alkali used:—5 c.c. of 0·5 N NaOH.
Time:—10 minutes.
Temperature:—17·5° C.
Blank titration:—38·68 c.c. of 0·05 N thiosulphate solution.

No.	Lævulose. Grms.	Back titration. Thiosulphate (0·05 N) solution. c.c.	Iodine (0·05 N) solution reduced. c.c.	Less amount due to dextrose. c.c.	Iodine per 1 grm. of lævulose. Grm.
1	0	25·40	13·28	—	—
2	0·25	24·97	13·71	0·43	0·0109
3	0·5	24·77	13·91	0·63	0·0080
4	2	23·63	15·05	1·77	0·0056
5	3	23·07	15·61	2·33	0·0049

(*d*) *Effect of Time of Reaction.*—As with sucrose, time appears to be an important factor in the extent to which lævulose is oxidised. Two grms. quantities of lævulose were allowed to react for 10, 20 and 30 minutes, under the standard procedure. The iodine reduction, which stood at 0·0087 after 10 minutes, had increased to 0·0154 in 30 minutes, but with a steady falling off in rate, tending to a maximum of, perhaps, 0·02 (Table XX.).

TABLE XX.—LÆVULOSE. EFFECT OF TIME OF REACTION.

Amount of lævulose in each case:—2 grms.
Amount of alkali used:—5 c.c. of 0·5 N NaOH.
Temperature:—17·5° C.
Blank titration:—39·31 c.c. of 0·05 N thiosulphate solution.

No.	Time. Minutes.	Back titration. Thiosulphate (0·05 N) solution. c.c.	Iodine (0·05 N) reduced. solution c.c.	Iodine per 1 grm. of lævulose. Grm.
1	10	36·57	2·74	0·0087
2	20	35·37	3·94	0·0125
3	30	34·45	4·86	0·0154

(*e*) *Iodine Value of Lævulose.*—Contrary to what is required in the case of sucrose, the point of analytical interest in connection with lævulose is its iodine value in small amounts, approximately equal to the amounts of dextrose likely to be titrated, since in analytical work this is by far the most frequent case. Sucrose, on the other hand, may occur in much greater quantity, and its effect has, therefore, been investigated over a wide range.

If the figures for lævulose in presence of dextrose (Table XIX.) be plotted as a curve, it is found that the iodine value when the lævulose is about 0·06 grm. is approximately 0·012. For solutions of invert sugar, etc., where about 0·12 grm. of invert sugar would be titrated per 40 c.c. of 0·05 N iodine solution, the effect of the lævulose on the iodine must be reckoned as 0·012 grm. of iodine per

1 grm. of lævulose. This is very much less than that noted by Miss Judd or by Baker and Hulton, but it is not altogether negligible. It is sufficient to cause errors amounting to nearly 1 per cent. in the dextrose if not taken into account.

V. INVERT SUGAR.

It now became of interest to examine how far the results established for dextrose and lævulose could be used for the analysis of mixtures containing invert sugar or, by suitable inversion procedure, sucrose. As shown above, the extent of oxidation of lævulose is modified by the presence of dextrose, and the only point of interest here is the behaviour when the amounts of the two sugars are approximately equal.

(*a*) *Iodine Value of Lævulose in Presence of an Equal Amount of Dextrose.*—By applying the method of extrapolation to the results for fairly large quantities of lævulose in presence of 0·06 grm. of dextrose (Table XIX.), it has already been shown that the iodine value of the lævulose when 0·06 grm. of this is present would be approximately 0·012. The apparent iodine value of the total sugar, therefore, would be

$$\frac{1{\cdot}410 + 0{\cdot}012}{2} = 0{\cdot}711.$$

In order to test this figure a solution containing 0·2 per cent. each of the pure recrystallised dextrose (previously dried) and lævulose was prepared and carefully measured out from a burette in a series of increasing quantities, containing from 0·04 to 0·16 grm. of mixed sugars. These solutions were titrated by the standard procedure, and the results obtained are given in Table XXI.

TABLE XXI.—INVERT SUGAR. IODINE VALUE OF MIXTURES OF EQUAL QUANTITIES OF DEXTROSE AND LÆVULOSE.

Amount of alkali in each case:—5 c.c. of 0·5 *N* NaOH.
Time:—10 minutes.
Temp.:—17·5° C.
Blank titration:—39·52 c.c. of 0·05 *N* thiosulphate solution.

No.	Dextrose. Grm.	Lævulose. Grm.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of mixed sugars. Grm.
1	0·0199	0·0199	35·05	4·47	0·712
2	0·0350	0·0350	31·67	7·85	0·712
3	0·0500	0·0500	28·32	11·20	0·711
4	0·0651	0·0651	24·98	14·54	0·709
5	0·0800	0·0800	21·73	17·79	0·706

The iodine value found for the mixed sugars was 0·712 for the smaller amounts, with a slight decrease with increasing amounts of sugar down to 0·706 for 0·16 grm. of sugars. Apparently the higher figures are due to the increase in the oxidation of lævulose as the dextrose diminishes. The rather low value for No. 5 may be due to a considerable diminution in the lævulose oxidation as the limit of excess of iodine is approached.

(*b*) *Iodine Value of Invert Sugar from Sucrose.*—A solution was next prepared by dissolving 1·900 grms. of the pure sucrose before used in 150 c.c. of water, adding 30 c.c. of 0·5 *N* hydrochloric acid, and boiling for 1 minute. The solution was then quickly cooled, almost neutralised with 0·5 *N* sodium hydroxide solution, and made up to 500 c.c. (See Bolton and Revis, *Fatty Foods*, p. 316.) A series of quantities was measured from a burette, as above, and titrated. A series of iodine values was obtained which were all about 0·010 higher than the corresponding value found for mixed lævulose and dextrose. (Compare Table XXII. with XXI.) Apart from this, the slight decrease from the lower to the higher amounts of sugar corresponds closely to that shown in Table XXI., and probably has the same explanation. The higher figures for this series suggested the possibility of the presence of substances of higher reducing power than the sugars, which could only be derived from the method of inverting the sucrose. In order to test this point, a solution of 0·2 grm. each of dextrose and lævulose was prepared and treated by the same method used for inverting the sucrose (boiling with acid for 1 minute, etc.) The solution was made up to 200 c.c., and measured portions titrated as before. The series (Table XXIII.) showed a gradation corresponding well with those of the previous series, and with indications of very slightly higher values than those of Table XXI., though not approaching those of Table XXII.

TABLE XXII.—INVERT SUGAR. IODINE VALUE OF INVERT SUGAR PREPARED BY BOILING SUCROSE SOLUTION WITH DILUTE ACID.

Standard procedure.
Blank titration:—39·52 c.c. of 0·05 *N* thiosulphate solution.

No.	Invert Sugar. Grm.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of invert sugar. Grm.
1	0·0404	34·92	4·60	0·723
2	0·0699	31·57	7·95	0·722
3	0·1001	28·15	11·37	0·721
4	0·1296	24·80	14·72	0·720
5	0·1600	21·46	18·06	0·716

TABLE XXIII.—INVERT SUGAR. IODINE VALUE OF MIXTURES OF EQUAL QUANTITIES OF DEXTROSE AND LÆVULOSE (AFTER BOILING WITH DILUTE HCl).

Standard procedure.
Blank titration:—39·50 c.c. of 0·05 *N* thiosulphate solution.

No.	Mixed sugar. Grm.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of sugars. Grm.
1	0·0402	34·98	4·52	0·714
2	0·0403	34·96	4·54	0·715
3	0·0397	35·03	4·47	0·715
4	0·0701	31·64	7·86	0·712
5	0·1002	28·26	11·24	0·712
6	0·1302	24·92	14·58	0·711
7	0·1599	21·68	17·82	0·707

(*c*) *Effect of Boiling Dilute Hydrochloric Acid on Invert Sugar Solutions.*—A more drastic treatment was then followed by dissolving 0·0801 grm. each of dextrose (dried) and lævulose in 150 c.c. of water, adding 30 c.c. of 0·5 *N* hydrochloric acid, and boiling for 45 minutes. The solution was cooled, neutralised, and the whole titrated by the standard procedure. The iodine consumption was 39·51 −21·11 = 18·40 c.c. of 0·05 *N* solution. This gives 0·729 grm. of iodine per grm. of mixed sugars—a value considerably higher than those of Table XXI.

In a final experiment to emphasise this effect of boiling with acids, 0·76 grm. of sucrose was dissolved in 150 c.c. water, 30 c.c. of 0·5 *N* hydrochloric acid added, and the solution boiled for 2 hours, then cooled, neutralised, and made up to 200 c.c. Measured portions were titrated, with the results shown in Table XXIV. These figures point to the presence of considerable quantities of substances other than the sugars in this solution, the only alternative being the unlikely conversion of lævulose into dextrose. The latter possibility was disposed of by estimation of reducing sugar by copper reduction; this showed that only 94·8 per cent. of the original invert sugar remained.

Hence it appears certain that the boiling of invert sugar solutions with dilute hydrochloric acid, such as used for inversion of sucrose, results in the destruction of the hexoses, with formation of products which reduce alkaline iodine. Even boiling for so short a period as 1 minute is sufficient to produce an appreciable amount of these substances.

TABLE XXIV.—INVERT SUGAR. APPARENT IODINE VALUE OF INVERT SUGAR BOILED WITH DILUTE ACID FOR 2 HOURS.

Standard procedure.
Blank titration:—38·52 c.c. of 0·05 *N* thiosulphate solution.

No.	Invert sugar. Grms.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of invert sugar. Grm.
1	0·0402	33·51	5·01	0·791
2	0·0795	28·72	9·80	0·782
3	0·1202	23·85	14·67	0·774
4	0·1603	19·13	19·39	0·763

(*d*) *Conditions for Satisfactory Inversion of Sucrose Solutions.*—In order to eliminate the destructive effect of the acid as much as possible, an inversion at a much lower temperature was tried. Ten c.c. of approximately 7 *N* hydrochloric acid were added to 80 c.c. of the sugar solution, and the flask allowed to stand in an incubator at 36° C. for 4 hours. The flask was then cooled, the solution almost neutralised with dilute sodium hydroxide solution, and made up to 200 c.c. Measured portions gave the figures shown in Table XXV., which agree well with those of Table XXI. (Mixtures of dextrose and lævulose.)

TABLE XXV.—INVERT SUGAR. IODINE VALUE OF INVERT SUGAR PREPARED BY INVERSION AT 36° C.

Standard procedure.
Blank titration:—39·45 c.c. of 0·05 *N* thiosulphate solution.

No.	Invert sugar. Grms.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. of invert sugar. Grm.
1	0·0400	34·97	4·48	0·711
2	0·1000	28·23	11·22	0·712
3	0·1599	21·58	17·87	0·709

Since a four hours' inversion would be unsuitable for many analytical purposes, an attempt was made to quicken the process by carrying out the inversion at 60° C. for 10 minutes. This gives practically the procedure used in the neutral polarisation process (Jackson and Gillis, *Bureau of Standards, Scientific Paper*, No. 375), except that sodium hydroxide is used for neutralisation of the acid, instead of ammonia.

The results are recorded in Table XXVI., and are, within experimental limits, in agreement with those of the dextrose and lævulose mixtures. The low value for No. 4 again points to the fact that the limit of excess of iodine is being approached. Under these conditions of inversion, then, and using about 0·12 grm. of invert sugar per 40 c.c. of 0·05 *N* iodine solution, the iodine value of invert sugar appears to be 0·710, a value which is in close agreement with that anticipated from a consideration of the behaviour of the individual sugars in other than equal proportions.

TABLE XXVI.—INVERT SUGAR. IODINE VALUE OF INVERT SUGAR PREPARED BY INVERSION AT 60° C.

Standard procedure.
Blank titration:—38·53 c.c. of 0·05 *N* thiosulphate solution.

No.	Invert sugar. Grm.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per 1 grm. invert sugar. Grm.
1	0·0398	34·05	4·48	0·714
2	0·0802	29·52	9·01	0·713
3	0·1198	25·12	13·41	0·710
4	0·1603	20·76	17·77	0·704

VI. ESTIMATION OF SUCROSE BY THE IODIMETRIC PROCESS.

It has now been satisfactorily established that the iodine reduction of invert sugar formed from sucrose under satisfactory condition is 0·710 grm. of iodine per grm. of sugar; and that the sucrose before inversion has a value of 0·003. Hence an increase in iodine reduction of 0·707÷0·95 will be noted per grm. of sucrose inverted. This gives a factor of 0·744 for calculating sucrose from the difference between "direct" and "invert" iodine reductions. In both titrations there should be present from 0·08 to 0·14 grm. of total iodine-reducing sugar, calculated as invert, for each 40 c.c. of 0·05 *N* iodine solution added.

VII. ESTIMATION OF INVERT SUGAR BY THE IODIMETRIC PROCESS.

From the experiments detailed above as to the effect of acid treatment on invert sugar solutions it will be evident that an accurate estimation of invert sugar iodimetrically is a difficult problem. In commercial and technical products containing invert sugar there may be varying quantities of the decomposition products affecting the iodine titration, and, unless the manner of preparation of the invert sugar is known, and a standard can be prepared under the same conditions (which is seldom the case), its estimation by this method may be by no means exact. Nevertheless, in the course of much work in this laboratory involving the analysis of invert sugar, sucrose mixtures, jams, etc., it has been found that the figures obtained under the standard conditions of the present report do give fairly satisfactory results for invert sugar. If any doubt is entertained as to the results being obtained for invert sugar in the routine examination of a particular class of product, an occasional check by the copper reduction method will at once show up any errors (as in the case of the experiments above, Table XXIV.).

VIII. THE USE OF WEAK ALKALIS IN PLACE OF SODIUM HYDROXIDE.

With a view to the elimination of the slight effects of lævulose and sucrose on the alkaline iodine solution, some experiments were carried out with weaker alkalis in place of the sodium hydroxide.

(*a*) *Sodium Bicarbonate.*—Ten c.c. of a 0·8 per cent. solution of the pure recrystallised dextrose were taken, diluted to about 50 c.c., and 40 c.c. of 0·05 *N* iodine solution added, followed by 10 c.c. of M/2 sodium hydrogen carbonate solution. The mixture was kept at 27·5° C. for 1 hour, and was then acidified and titrated. Only 10·41 c.c. of 0·05 *N* iodine solution had been consumed, corresponding to an iodine value for the dextrose of 0·826. Without further prolonging the time or raising the temperature to an inconvenient extent, it is evident that the complete oxidation of the dextrose could only be attained by using excessive amounts of the alkali, which for several reasons (*e.g.* effervescence upon neutralisation) might be objectionable.

(*b*) *Sodium Carbonate.*—Ten c.c. of a 0·8 per cent. solution of the dextrose were used, and treated with 40 c.c. of iodine solution and 5 or 10 c.c. of 0·5 *N* sodium carbonate solution for half-hour periods at 17·5° C. and 27·5° C. The results are given below (Table XXVII.).

TABLE XXVII.—USE OF SODIUM CARBONATE AS ALKALI.

0·08 grm. dextrose in each case. Standing for ½ hour.

No.	Blank titration. Thiosulphate (0·05 *N*) solution. c.c.	Amount of 0·5 *N* sodium carbonate. c.c.	Temp. ° C.	Back titration. Thiosulphate (0·05 *N*) solution. c.c.	Iodine (0·05 *N*) solution reduced. c.c.	Iodine per grm. of dextrose. Grm.
1	39·30	5	27·5	26·73	12·57	0·997
2	39·30	10	27·5	21·64	17·66	1·401
3	39·30	10	17·5	21·95	17·35	1·376

These figures were sufficient to show that, with suitable conditions, a satisfactory result could probably be obtained for dextrose by the use of sodium carbonate.

Before proceeding further, however, it was thought advisable to determine to what extent the action on lævulose would take place. Two grms. of lævulose were dissolved, 40 c.c. of 0·05 *N* iodine solution and 10 c.c. of 0·5 *N* sodium carbonate solution added, and the mixture kept for ½ hour at 27·5° C. (corresponding to No. 2 of Table XXVII.); a blank was carried out at the same time. The difference between the back titrations amounted to 3·96 c.c. of 0·05 *N* thiosulphate solution, giving an iodine reduction of 0·013 grm. per 1 grm. of lævulose. Hence it appears that for the conditions likely to give a satisfactory result for dextrose, the iodine reduction of the lævulose is just as marked whether sodium carbonate or sodium hydroxide be the alkali used. Cajori used sodium carbonate to the extent of about 1 per cent. in the mixture, with room temperature of about 20° C., and obtained satisfactory results for dextrose. Such amounts of sodium carbonate, in view of the present experiments, might be expected to have an appreciable action on the lævulose. In Cajori's experiments, however, the lævulose used was insufficient to show any effect on the iodine reduction within the limits of experimental error.

There thus appeared to be no advantage in the use of sodium carbonate, and the necessity for a higher temperature or longer time of reaction placed it at a considerable disadvantage as compared with sodium hydroxide. Accordingly the investigation of its effects was not carried further.

(*c*) *Borax.*—A number of titrations were made, with the use of dextrose and iodine as before, in conjunction with 0·25 *N* borax solution. (The 0·5 *N* solution crystallised, and had to be diluted.) In the following summary of the iodine values obtained, the amount of alkali is expressed as 0·5 *N* solution:

TABLE XXVIII.—IODINE VALUES FOR DEXTROSE WITH BORAX AS ALKALI.

No.	Amount of borax (0·05 N) solution. c.c.	Time.	Temp. ° C.	Iodine value found.
1	5	10 mins.	17·5	0·475
2	5	2 hrs.	17·5	1·201
3	5	½ hr.	27·5	1·225
4	5	1 „	27·5	1·349
5	10	1 „	27·5	1·264
6	4	2 hrs.	27·5	1·365
7	6	2 „	27·5	1·395
8	7	2 „	27·5	1·380
9	10	2 „	27·5	1·350
10	6	20 mins.	50° C.	1·409

Here, again, a satisfactory result for dextrose can be obtained, but only by using a fairly high temperature or a sufficient length of time. It was observed that the blank estimations (made side by side with each dextrose titration) were not in close agreement at 27·5° C., differences amounting to as much as 0·2 c.c. being found, which was a much greater difference than any found at ordinary temperatures. This adds still further to the objections to the use of weak alkalis, such as borax, which require an elevated temperature for the reaction. The only justification for its use would seem to be the smallness of the effect on lævulose, 2 grms. of which after 1 hour at 27·5° C., with the use of 5 c.c. of 0·5 N borax solution, showed an iodine value of only 0·0005.

(*d*) *Mixed Borax and Sodium Hydroxide.*—A mixed 0·5 N solution of borax and sodium hydroxide was prepared, with 2 equivalents of borax to one of sodium hydroxide (*i.e.* 60·3 grms. borax and 6·7 grms. of sodium hydroxide per litre). This solution was tested as in the case of the borax alone.

TABLE XXIX.—IODINE VALUE OF DEXTROSE WITH THE USE OF MIXED BORAX AND SODIUM HYDROXIDE.

No.	Amount of alkali (0·5 N) solution. c.c.	Time. Hours.	Temp. ° C.	Iodine value found.
1	4	1	27·5	1·355
2	5	1	„	1·380
3	6	1	„	1·378
4	7	1	„	1·369
5	5	2	„	1·400

The complete oxidation could, no doubt, be obtained with this mixture, but there appears to be no advantage over the use of borax alone, and the effect on lævulose would probably be more pronounced.

SUMMARY.

(*a*) Certain discrepancies in the literature on the subject as to the reduction by sugars of iodine in alkaline solution have been explained.

(*b*) The effects of proportions of iodine and of alkali used, and of time and temperature, in the cases of dextrose, lactose, sucrose, and lævulose have been examined.

(*c*) Dextrose and lactose are quantitatively oxidised to monobasic acids under suitable conditions. The action on sucrose and lævulose is small and very subject to the conditions of experiment, but, under definite conditions, these sugars exert a constant reducing power.

(*d*) A standard procedure has been outlined, which consists in using about 50 c.c. of solution containing about 0·08 grm. of dextrose or its equivalent, adding 40 c.c. of 0·05 *N* iodine solution and 5 c.c. of 0·5 *N* sodium hydroxide solution, and allowing the mixture to stand for 10 minutes at 17·5° C., then acidifying it with 5 c.c. of 2 *N* sulphuric acid, and at once titrating the excess iodine with 0·05 *N* thiosulphate solution.

(*e*) The iodine values found under these conditions are:

Dextrose	1·410	grms. iodine per 1 grm. sugar.
Lactose hydrate	0·705	,, ,, ,, ,, ,,
Sucrose	0·003	,, ,, ,, ,, ,,
Lævulose (in large amount)	0·0065	,, ,, ,, ,, ,,
Lævulose (in presence of equal amounts of dextrose)	0·012	,, ,, ,, ,, ,,

(*f*) Sucrose can be estimated by an iodine titration before and after inversion, with an accuracy of about 0·2 per cent., provided the inversion is carried out at a temperature not exceeding 60° C. The iodine value for sucrose is then 0·744.

(*g*) Invert sugar can be estimated in many products in the absence of lactose (and maltose) by a single titration with iodine, with sufficient accuracy for technical control purposes. The iodine value may be taken as 0·710.

(*h*) Weaker alkalis, such as sodium carbonate or borax, when used in place of sodium hydroxide, necessitate a much longer time of reaction or a higher temperature, and are therefore less suitable for use than sodium hydroxide.

(*Note.*—While the above work was being carried out, two papers have been published by I. M. Kolthoff (*Zeitsch. Untersuch. Nahr. Genüssm.*, 1923, **45**, 131, 141) on the iodimetric estimation of sugars. The conditions arrived at for the estimation of dextrose and lactose, with the use of sodium hydroxide for rendering the liquid alkaline, are very close to those found here. A few experiments on the reducing effect of sucrose and lævulose are described, and, so far as the results go, they are approximately in accordance with those of this report. The susceptibility of the reducing effect of lævulose to time, temperature, etc., is, however, not worked out; consequently the conditions to be used in applying the suggested correction for the presence of lævulose in estimating the dextrose of invert sugar are inadequately defined.)

This investigation was carried out in the laboratories of the British Association of Research for the Cocoa, Chocolate, Sugar, Confectionery, and Jam Trades. The larger portion of the experimental work was done by J. R. Heather.

DISCUSSION.

Mr. **CHARLES M. CAINES** enquired whether the authors' method was applicable to the accurate estimation of small quantities of sugar in blood and similar biological material. He mentioned that at the meeting of the British Medical Association at Portsmouth he was demonstrating the method recommended by Dr. Maclean for the estimation of sugar in the blood in connection with the new insulin treatment. While listening to the author, it had struck him that if the process were capable of accurately estimating minute quantities of sugar it might afford a useful modification of the Maclean method, which, at present, depended on the reduction of alkaline copper iodine solution and involved a full six minutes' boiling.

Mr. A. E. **PARKES** enquired whether the authors' method could be applied to mixtures of sugars, such as lactose, invert sugar, and cane sugar in condensed milk, and glucose syrup, invert sugar and cane sugar in jams. After clarification with lead acetate, was it possible to obtain a solution which could be treated by the iodine process for the estimation of sugars?

Mr. C. L. **HINTON**, in reply, said the process was originally introduced by biochemists for the estimation of sugars in plants. He considered it might be equally useful for the estimation of sugar in physiological products, provided that proteins and other bodies which reacted with iodine were first removed.

As regards the question of its application to jams after clarification, they had found no difficulty in using the process for ordinary jams or for invert sugars and lactose, but glucose rather complicated matters. One could use the process in conjunction with polarisation, but, if lactose and glucose were both present, the estimation became more complicated, and it was necessary to use fermentation methods.

As regards sucrose, the accuracy was equal to that of the Fehling method, and the process was very much more rapid.

The Gold-Beater's Skin Test for Tannins.

By PHYLLIS HONOR PRICE, B.Sc.

(*Read at the Meeting, November* 7, 1923.)

In a recent communication from this laboratory (Atkinson and Hazleton, *Biochem. J.*, 1922, **16**, 516; Analyst, 1923, **48**, 38) a qualitative test for tannins was described which is based on the fixation of the tannins on gold-beater's skin and the subsequent staining with ferric chloride. A specific test which demonstrates the " anning" properties of a tannin is of the greatest importance to the chemistry of the tannins, especially at a time when the syntheses of "tannins" have become the fashion, and I have therefore, at the suggestion of Dr. Nierenstein, attempted to increase the delicacy of the test, with the result that I have elaborated a method capable of detecting the "tanning" properties of 0·00005 grm. of gallotannin in 1 c.c. of water.

The gold-beater's skin test, as originally described by Atkinson and Hazleton, involves four distinct operations, namely (1) the swelling of the gold-beater's skin so as to make it permeable to the tannins; (2) the tanning of the gold-beater's skin; (3) the washing of the tanned gold-beater's skin so as to remove any tannin which had not been fixed; and (4) the staining of the tanned gold beater's skin with ferric chloride. I have studied all these operations separately, and, they are referred to in the present communication as *swelling*, *tanning*, *washing* and *staining*, respectively. It was also observed by Atkinson and Hazleton that the tanned and subsequently stained gold-beater's skin may be decolorised with dilute hydrochloric acid. I have used the latter process as a method for the detection of "phlobaphenes," and is referred to in this communication as *decolorising*.

Swelling*.—Atkinson and Hazleton use water as a swelling reagent and, as the gold-beater's skin prepared according to their directions is frequently unevenly stained, I have tried other swelling reagents. I find an evenly stained specimen is always obtained if 2 per cent. hydrochloric acid is used for this purpose. As a comparison with 2 per cent. hydrochloric acid (about half-normal) the swelling properties of normal, half-normal and deci-normal solutions of sulphuric acid, acetic acid, succinic acid, formic acid, tartaric acid, and oxalic acid were also tried. None of these proved as effective as a 2 per cent. solution of hydrochloric acid. Similarly, stronger reagents such as for example, 5 per cent. hydrochloric acid, cannot be recommended as they, by themselves, darken the colour of the untanned gold-beater's skin and thus subsequently mar the staining effects produced by ferric chloride.

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* These experiments were carried out in collaboration with Miss R. A. E. Colborn.

TANNING.—It must be noted that if hydrochloric acid is used as a swelling reagent it is very important to wash the gold-beater's skin carefully before tanning, as any hydrochloric acid, if present, affects the colour produced by ferric chloride. This is, of course, unnecessary when water is used as a swelling reagent.

Atkinson and Hazleton have fixed 30 minutes as the maximum time required for the tanning of the gold-beater's skin. I find that this suffices even for a solution of 0·00005 grm. of gallotannin in 1 c.c. of water.

WASHING.*—Atkinson and Hazleton have fixed the time required for the washing of the tanned gold-beater's skin as two minutes at a constant drip of two drops per second. From a large number of experiments I have come to the conclusion that the washing must be prolonged to 15 minutes, especially when ferrous sulphate and ferrous chloride are used as staining reagents. As will be seen later, these iron salts are far more sensitive than ferric chloride, and they were found to stain even gold-beater's skin which had been treated with gallic acid, if only washed for 2 minutes as recommended by Atkinson and Hazleton. This, however, is not the case if the washing is carried out for 15 minutes at a constant drip of 2 drops per second.

STAINING.—Numerous qualitative tannin tests have been recommended from time to time (compare, for example, Grasser, *Handbuch für Gerberei-Chemische Laboratorien*, 1922, pp. 241–273). I have, in addition to different iron-salts, also tested most of the heavy metals which have been suggested and several aniline dyes. In addition to this, I have tried the nitrous ether test of Vinson (*Botanical Gazette*, 1910, **49**, 222). All the reagents were at first tried on the untanned gold-beater's skin. Those which stained the skin without previous tanning were discarded. The following reagents were found to affect the untanned gold-beater's skin:

(1) Ferric oxalate.

(2) Silver nitrate, titanous chloride, titanic chloride, uranium acetate, gold chloride and thorium nitrate.

(3) The following aniline dyes:

Methyl violet, nigrosine, brilliant green, methyl green, Congo red, methylene violet, malachite green, methylene blue, crystal violet, Victoria blue, and cotton blue.

Hæmatoxylin was also tried and acted similarly.

A 1 per cent. solution of the staining materials was used, except in the case of the aniline dyes, which were 0·1 per cent. solutions.

In this connection it is interesting to note that the following substances, although recommended as specific reagents for tannins, had practically no effect on the tanned gold-beater's skin: Mercurous chloride, phosphotungstic acid and thallium chloride.

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* These experiments were carried out in collaboration with Miss R. A. E. Colborn.

The iron-salts tested are herewith arranged according to their sensitiveness towards different gallotannin solutions, 1 per cent. solutions of the iron-salts being used:

Group I. *Sensitiveness*: 0·005 *per cent. gallotannin.*
Ferrous sulphate, ferrous chloride.

Group II. *Sensitiveness*: 0·01 *per cent. gallotannin.*
Ferric alum, ferric malate, ferric potassium tartrate, ferric sulphate, ferric ammonium chloride.

Group III. *Sensitiveness*: 0·02 *per cent. gallotannin.*
Ferric chloride.

Group IV. *Sensitiveness*: 0·05 *per cent gallotannin.*
Ferric perchloride, ferric acetate.

Group V. *Sensitiveness*: 0·1 *per cent. gallotannin and less.*
Ferrous ammonium sulphate, ferrous oxalate, ferric potassium oxalate, ferric succinate, ferric formate, ferric ammonium tartrate, ferric ammonium oxalate.

Mitchell's reagent (ANALYST, 1923, **48**, 2), which is a solution of 0·1 grm. of ferrous sulphate + 0·5 grm. of Rochelle salt in 100 c.c. of water was sensitive for 0·01 per cent. gallotannin and, consequently, can be grouped under II., although it contains less iron than the reagents given under Group II.

The heavy metals (1 per cent. solutions) used gave the following results:

Group I. *Sensitiveness*: 0·01 *per cent. gallotannin.*
Ammonium molybdate and vanadium chloride.

Group II. *Sensitiveness*: 0·05 *per cent. gallotannin.*
Potassium chromate.

Group III. *Sensitiveness*: *Less than* 0·1 *per cent. gallotannin.*
Copper sulphate.

As will be seen, the sensitiveness of these heavy metals is below that found for Group I. of the iron-salts.

Vinson's nitrous ether test gave very good results. Although amyl nitrite, when used on the gold-beater's skin in the same way as the other reagents, showed 0·01 per cent. gallotannin, it was possible to increase the sensitiveness to 0·005 per cent. by using the following technique:

The tanned skins were placed on paraffin-wax in a covered petri dish, which also contained a watch-glass with 1 c.c. of amyl nitrite. The solution being very volatile, the dish was soon filled with the fumes. No effect was observed until after about 20 minutes. Eventually, after repeated experiments, 3 hours was chosen as the time for the fumes to react, giving a deep brown-yellow coloration.

Similar experiments in which ethyl nitrite was used were found to detect only 0·1 per cent. of gallotannin.

In connection with all the experiments mentioned under staining it must be noted that control tests in which gallic acid was used were made in every case. None of them showed the slightest staining effect on gold-beater's skin treated with gallic acid.

DECOLORISING.*—Atkinson and Hazleton found that tanned and subsequently stained gold-beater's skin may be decolorised with 2 per cent. hydrochloric acid. I have obtained most satisfactory results when using 5 per cent. hydrochloric acid. In addition to this, I have also tested the decolorising effect of the following acids:

Oxalic acid—completely decolorised.
Tartaric acid—only very slightly decolorised.
Formic acid—not completely decolorised.
Succinic acid—not completely decolorised.
Sulphuric acid—completely decolorised.
Acetic acid—only very slightly decolorised.

During these experiments it was noticed that whereas pyrogallol tannins give a completely decolorised gold-beater's skin, on treatment with hydrochloric acid, catechol tannins leave an orange-brown stain on the skin, probably due to phlobaphenes. A series of experiments with a number of catechol tannins showed it to be a general reaction, and this was also confirmed by adding catechol tannins to pyrogallol tannins. I would therefore suggest that this method be used as a Test for Phlobaphenes.

GENERAL TECHNIQUE.—A small piece of gold-beater's skin, about ½ inch long and ¾ inch wide, is pinned on a flat surface of paraffin-wax, which is prepared by pouring melted paraffin-wax into a watch-glass.

1. *Swelling.* One c.c. of a 2 per cent. solution of hydrochloric acid is pipetted on to the skin and left standing for 10 minutes. The skin is then washed with distilled water at a constant drip of two drops per second for two minutes.

2. *Tanning.* The skin is treated with 1 c.c. of the solution to be investigated for the presence of tannin for 30 minutes. It is then washed as before for 15 minutes.

3. *Staining.* One c.c. of a 1 per cent. solution of either ferrous sulphate or ferrous chloride is left standing on the skin for 15 minutes and, as before, the skin is washed for two minutes.

4. *Decolorising when testing for phlobaphenes.* One c.c. of a 5 per cent. solution of hydrochloric acid is left on the skin for 2 minutes, and then the skin is washed, as before, for 2 minutes.

When dry, the skin may be mounted for reference and compared with skins treated with gallotannin solutions of varying concentrations. When very minute quantities indeed of tannins are suspected to be present, it is advisable to compare the skin also with untanned stained skins and stained skins which have been treated with gallic acid.

BIOCHEMICAL LABORATORY, CHEMICAL DEPARTMENT,
UNIVERSITY OF BRISTOL.

* These experiments were carried out in collaboration with Miss E. S. Smyth, B.Sc.

DISCUSSION.

Mr. W. PARTRIDGE, mentioning a plant which he had sent to Mr. Mitchell in connection with the paper, said that tannins in land plants had more or less been investigated, but that water plants had been neglected in this respect. When approached on the matter he had happened to know one particular water plant—a species of *Potamogeton*—which contained tannin. This plant, which rotted in the water and produced an unpleasant smell, was found only in certain lakes; it had been observed that water taken from those lakes was quite inky, and, on investigation, it had seemed that this particular weed, in association with iron derived from ferruginous clay in the bed of the lake or from the iron pipes, was to blame.

Dr. H. E. Cox remarked that the difference between the specimen of untanned skin and one of the specimens of tanned skin seemed to be scarcely distinguishable by artificial light, and suggested that perhaps a greater difference was evident by daylight.

Mr. G. RUDD THOMPSON said he was much interested in the originality of the work done; but, referring to the manner of mounting the specimens, thought the colours would have been more easily compared if the specimens had been arranged so that one could look through the gold-beater's skin; by artificial light it was very difficult to see any appreciable difference in colour between the treated and untreated skin in the case of some of the specimens shown mounted on card.

Mr. W. T. BURGESS pointed out that the pins used to hold the goldbeater's skin to the paraffin wax during treatment might affect the colour; he would like to have particulars of the pins used.

Mr. C. A. Mitchell, replying on behalf of the author, said he had tried the method, but could not say he was at all satisfied with the technique. In his opinion, the acid was liable to attack the pins and the wax was porous at the various perforations. In his experiments, therefore, he had fixed the skin between an ordinary microscope slide and a perforated one, held together by two screw clips, and had found this most satisfactory. It also enabled the treated skin to be examined by transmitted light. He had applied the test to a 5 per cent. extract of Mr. Partridge's pond weed, which gave a black coloration with ferric chloride on the skin.

The author's method of mounting her specimens gave a good colour by daylight. He had tried the effect of "osmic acid" as a staining reagent in the test, and had found it to give excellent results.

Mr. RUDD THOMPSON, referring to the arrangement with perforated slides devised by Mr. Mitchell for mounting his specimens, said that it might also be adopted with very favourable results in a microspectroscope; the cell made by fixing a short length of barometer tube to a plain slide often became detached in the middle of the operation.

The Corrosion of Aluminium Cooking Utensils.

BY C. KENNETH TINKLER, D.Sc., F.I.C., AND HELEN MASTERS, B.Sc.

IN the *Lancet* of January 4th, 1913, an account was given of some work carried out in the *Lancet* laboratory entitled "Some Kitchen Experiments with Aluminium." The general conclusion was reached "that any suspicion that aluminium may communicate poisonous qualities to food in the process of cooking may safely be dismissed."

Although, as a result of experiments carried out since 1913, there appears to be no reason to doubt the correctness of the above conclusion, yet in view of the greatly extended use of aluminium for cooking purposes, an account of some experiments we have made with aluminium cooking utensils may be of interest. Accounts of experiments of various other investigators on the corrosion of aluminium are to be found in the *Journal of the Institute of Metals*.

It is well known to those who make use of such vessels for cooking purposes that in certain cases extensive corrosion appears to have taken place, as indicated by the discoloration produced on the vessel. Thus when tap water is boiled in an aluminium saucepan a very dark stain is often produced, which is, however, much more pronounced in some cases than in others. The stain produced varies with the nature of the water and of the aluminium employed. Stains of different intensities are also often obtained with the same vessel and the same kind of water under different conditions of heating, and in some cases the stain may be partly masked owing to the deposition of calcium carbonate when hard water is boiled.

It is also apparently well known that this stain is removed if the vessel is subsequently used in an operation in which an acid liquid is employed, as for example in stewing fruit, and it can also be removed if a strongly alkaline liquid is heated in the vessel, or by means of an abrasive.

If water containing substances in solution or suspension is heated in an aluminium saucepan it is often found that the stain produced is much less pronounced than if water alone is heated in it. Thus if an egg is "boiled" in water in such a vessel a dark stain may be produced on the metal, whereas if the same vessel is used for "scrambling" an egg no stain is produced. It would be expected that if the egg were in any way connected with the apparent corrosion the attack would be more pronounced in the second case. Since, however, aluminium may be dissolved with or without blackening, it is impossible simply from the appearance of the saucepan to decide in which case greater corrosion has taken place.

THE NATURE OF THE DARK STAIN.—There is apparently no doubt that the dark stain is due to the presence of impurities in the aluminium, the chief of which is iron; and that by the action of an alkaline water an extremely small amount of aluminium is removed from the surface, leaving behind the iron and other impurities which are insoluble in the alkaline water.

Iron is, of course, readily soluble in acid, so that the dark stain is removed by an acid liquid, and no stain is produced when commercial aluminium, which contains iron impurities, is being dissolved in acid.

We have compared the amount of iron removed from a stained and from a bright aluminium saucepan by treatment with very dilute sulphuric acid, and find approximately twice as much in the case of the stained pan.

In the case of one sample of commercial aluminium which showed pronounced blackening with tap water, an insoluble residue amounting to 2·2 per cent. of the material was obtained when the metal was dissolved in a solution of sodium hydroxide, this residue being a finely divided black powder, whilst for the same material the amount insoluble in hydrochloric acid was only 0·38 per cent. When such a sample of aluminium is dissolved in sodium hydroxide solution no dark film is observed as solution proceeds, although one may appear at first, as in this case probably owing to the rapid solution of the aluminium, the particles of iron become detached and remain suspended in the solution. This explains the removal of the dark stain by strong alkali.

For a given sample of aluminium and different samples of natural waters the intensity of the stain appears to depend in most cases, on the hydroxyl ion concentration of the water; the greater this concentration the darker the stain.

THE PROTECTION OF ALUMINIUM FROM CORROSION.—In view of the results obtained by Friend (*Trans. Chem. Soc.*, 1921, 932) on the protection of iron from corrosion in the presence of colloids, we carried out a number of experiments with reference to the protection of aluminium. There is apparently no doubt that the colloidal matter, which is frequently present when aluminium vessels are used for cooking food, materially lessens the amount of aluminium dissolved. Thus in one experiment the loss in weight of a disc of aluminium heated in a dilute solution of agar in tap water was only one-third of the loss in weight produced when the same disc was heated in tap water alone, and only very slight darkening was observed in the first experiment. The non-formation of a stain when an egg is "scrambled" in an aluminium saucepan may also be due to the presence of colloidal matter. Some results on the protection of aluminium by colloids are given by Friend (*loc. cit.*, 1922, 468.)

We have found that it is possible to prevent the discoloration of aluminium by tap water. If a solution of potassium dichromate is boiled in an aluminium saucepan no blackening takes place; in fact, aluminium so treated shows a slight gain in weight and is rendered "passive." Treatment with tap water does not now produce a stain. The effect, however, is not permanent.

To illustrate this action, a small quantity of a solution of dichromate is heated for some time in a bright aluminium saucepan, the pan is then washed out, filled with tap water and heated. A well-defined stain is obtained above the original level of the dichromate solution, but the part previously covered by the solution remains quite bright.

In connection with the cleaning of aluminium, reference should be made to a paper by Seligman and Williams (*J. Inst. Metals*, **1922**, 297; ANALYST, **1922**, **47**, **493**). They find that the attack of an alkaline solution on aluminium is considerably retarded in the presence of sodium silicate, and that commercial preparations sold for cleaning the metal usually contain sufficient sodium silicate for this purpose.

CHEMICAL LABORATORY,
HOUSEHOLD AND SOCIAL SCIENCE DEPARTMENT,
KING'S COLLEGE FOR WOMEN, KENSINGTON, W.8.

Note.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE ESTIMATION OF PHOSPHINE IN ACETYLENE.

IN estimating phosphine in acetylene generated from commercial calcium carbide the method of Lunge and Cererkreutz (as given by V. B. Lewes in *Acetylene*) was employed. The method is designed to give:—(1) The weight of acetylene evolved; (2) the amount of phosphorus absorbed by the wash solution (sodium hypochlorite); (3) the sulphur in the filtrate from (2) by precipitation as barium sulphate.

When the estimation was carried out as described by Lewes, with the use of granular calcium chloride to dry the gas prior to absorption, but with the substitution of four wash-bottles for the absorption bulbs, the following anomalous results were obtained:

(1) After clearing out the acetylene the last two wash-bottles smelt strongly of phosphine;

(2) The phosphorus recovered increased progressively in amount from the first to the last wash-bottle;

(3) Free sulphur was immediately precipitated in the first wash-bottle, but not in the succeeding bottles.

The sodium hypochlorite wash solution was prepared by the interaction of bleaching powder and sodium carbonate and made up to three per cent. NaOCl. A second solution was made by the method employed by L. M. Dennis and W. J. O'Brien (*J. Ind. Eng. Chem.*, **1912**, **834**), in which chlorine gas is passed into sodium hydroxide solution to saturation, excess of chlorine removed with a current of air, and the solution made up to 3 per cent. Both solutions gave identical results. The solutions were then tested by passing hydrogen containing phosphine through them, and it was observed that throughout the experiment the smell of phosphine persisted in the hydrogen issuing from the last bottles. The amount of phosphorus recovered again increased progressively in amount from the first to the last bottle of each series.

Another possible source of error is the use of calcium chloride in drying the acetylene, for, from an investigation by Stock, Böttcher and Lenger (*J. Chem. Soc.*, **1909**, **A ii**, **727**), it appears that inflammable P_2H_4 may be converted into the solid $P_{12}H_6$ by passing over granular calcium chloride, the solid $P_{12}H_6$ being retained by the chloride.

A modification of the combustion method was finally used. In order to avoid the use of oxygen and a special burner the acetylene was led either direct from the generator or from a gas-holder to an ordinary bicycle lamp acetylene burner. The flame was almost entirely enclosed in a glass hood connected directly with four wash-bottles containing slightly ammoniacal distilled water, and a rapid current of air was drawn through the system. By adjustment of the supply of acetylene the flame was prevented from smoking and the rate of the air current was such as to ensure complete withdrawal of the products of combustion. No oxygen supply was required. It was found that the phosphorus was retained almost wholly in the first wash-bottle, with a trace in the second and none in the third and fourth. The total recovered was nearly ten times as great as was obtained by the method of Lunge, though still within the limits for commercial acetylene as given by Lewes.

T. E. PERKS.

THE DOMINION LABORATORY,
WELLINGTON, NEW ZEALAND.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

COUNTY BOROUGH OF SALFORD.

ANNUAL REPORT OF THE BOROUGH ANALYST FOR 1922.

THE total number of samples analysed was 1534,of which 1452 were taken under the Sale of Foods and Drugs Acts. Of these samples 82 (5·6 per cent.) were returned as adulterated, as compared with 8·7 per cent. in the previous year.

MILK.—Forty-nine of the 923 samples were adulterated. Of the 426 samples tested for dirt, 167 yielded no sediment; 59 gave 0·5 part, 117, 1· part; 4, 1·5 parts; 50, 3 parts; 6, from 4 to 5 parts; 14, 6 parts; 5, gave 9 parts; 2, 12 parts; and 2, 15 parts of dirt per 100,000. The lenient standard of a sediment not exceeding 5 parts per 100,000 has again been fixed as a maximum, and all milks giving a sediment in excess of that figure have been regarded as adulterated. The results obtained during the six years from 1915, however, indicate that a standard of not more than 1 part per 100,000 would be perfectly fair, and that milk containing 2 or more parts per 100,000 should be classified as unsatisfactory.

It is suggested that if the supply of milk of Grade "A" quality is so important that it is worth while having special legislation to ensure it, it would seem equally important to prevent the sale of grossly contaminated milk as ordinary milk.

MARGARINE.—It would seem that it was not the intention of the legislature that butter substitutes should be described as mixtures of butter and margarine, particularly in view of the definition of margarine in Sec. 13 of the Act of 1907 as "any article of food, whether mixed with butter or not, which resembles butter and is not milk-blended butter."

In view of these considerations proceedings were taken against four large firms for the improper labelling of margarine as mixed or blended with best butter.

Fines were inflicted in three cases, and the fourth was dismissed, as the word "margarine" was held to be the most conspicuous on the carton. As a result of these prosecutions the slips inserted under the statutory wrappers, and the cartons, were withdrawn from all the branch shops of the firms throughout the country.

COCOA.—Fourteen samples were examined, of which 3 (prepared by one manufacturer) contained one-tenth grain of arsenic (expressed as arsenious oxide) per lb. The local manager of the firm was notified as to the result, and withdrew all stocks of cocoa which had been delivered to shops in the Borough prior to August 1, 1922.

CANNED GOODS.—All of the 20 samples of canned goods (6 of fish, 11 of fruit, and 3 of jam) contained small quantities of tin in solution when turned out of the can and thoroughly mixed. A sample of canned "Melon and Orange" jam contained 6 grains of metallic tin per lb. The vendor was interviewed and undertook to destroy the remainder of the stock, and the facts were communicated to the Authority in whose district were the premises of the wholesale dealer.

DRUGS.—Of the 116 samples examined, only three were adulterated. One of these, Epsom salts, contained 20 parts of arsenic per million, and the other two, borax, each contained 50 parts of arsenic per million. The vendors were notified, and at once withdrew their stocks.

PRESCRIPTIONS.—Fifty-one samples were taken, of which only 3 were unsatisfactory.

SODA WATER.—Two of 4 samples were found to be devoid of added sodium bicarbonate. The manufacturers agreed that "soda water" sold in Salford should actually be *soda* water. In 1919 the local Association of Mineral Water Manufacturers gave the opinion that soda water should contain 10 grains of sodium bicarbonate per pint. It is generally admitted that any quantity between 5 and 10 grains per pint is a suitable standard.

G. D. ELSDON.

Meteorological Office, Air Ministry.

ADVISORY COMMITTEE ON ATMOSPHERIC POLLUTION.

REPORT ON OBSERVATIONS IN THE YEAR ENDING MARCH 31ST, 1922.*

THE STANDARD GAUGE.—The results obtained for the amount of atmospheric pollution with 22 gauges operated by 12 authorities in different parts of the country are dissected out in tables, comparisons with averages for the corresponding months of previous years being made for the first time. Instead of the results being expressed, as previously, in metric tons per square kilometre, these figures have been multiplied by 100 to avoid decimal points, and therefore can be read as metric tons per 100 sq. kilometre, or as weight in grms. of deposit over a 10 metre square. In the mean monthly values for the whole year the most notable feature is the universally low rainfall, and the nearly universally low pollution average, probably directly connected with the restricted use of coal owing to the coal strike which lasted from April 1st to July 4th.

Tar was above the average at 3 out of the 8 London stations, Newcastle and Southport; Southwark Park, London, coming first, with 143 metric tons per 100 sq. kilometre, against an average for the previous 5 years of 35 metric tons.

Insoluble Carbonaceous Matter other than Tar was above the average at 3 London stations, Hull, Liverpool, Newcastle, Rothamstead, and Southport, the greatest deposit being in London (1043 m.t., with Newcastle next, 992 m.t.).

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* M.O. 256. H.M. Stationery Office, Kingsway, W.C.2. Price 3s. net.

Insoluble Ash (*or dust*) was above the average at 4 London stations, Hull, Liverpool, Rothamstead and St. Helens, the greatest pollution again being in London (2138 m.t.), with Liverpool and Hull next (1600 and 1468 m.t. respectively).

Loss on Ignition (*volatile soluble compounds*) was only above the average at 2 stations, as was also the case with *Soluble Ash* (*non-volatile soluble compounds*).

Total Solids were greatest in London (3630 m.t.), and above the average in 3 stations.

The Total Deposit. On the average the summer total is less than the winter in 14 cases, equal in 3, and greater in 5, but in 1921–22 this was confirmed in only 8 cases, and contradicted by the relatively large deposits in 6 cases and low deposits at 2 stations.

A report by Dr. Ashworth on the *Deposits obtained at Rochdale* deals with the possible causes of the very high atmospheric deposit; with the seasonal variation shown by the total deposit; the close correspondence between the amount of soluble matter and rainfall; and the inverse relationship between the amounts of soluble and insoluble matter.

The Records of the Automatic Filter. Data for 4 stations, Kew Observatory, Richmond, Surrey; Savoy Hill, London; Westminster, London; and Rochdale, were available. In the case of the first 3 the results are treated on similar lines to those adopted in the 7th report (ANALYST, 1922, **47**, 256–258), the days being divided into summer and winter and the winter days into foggy (with a maximum shade number of 4 or over) and non-foggy. The days are further grouped into week-days (excluding Saturdays), Saturdays and Sundays. In considering the hourly distribution curves they are found on the whole to be very similar to those for previous years. In London on days without abnormal fog impurity begins to rise at 5 to 6 a.m.; attains a maximum at 10 a.m.; falls steadily till about 3 p.m.; rises slightly between 5 and 7 p.m.; and then falls, but on foggy days there is great delay in attaining the maximum. Taking the year as a whole, a slight improvement in the atmosphere is noted. The Rochdale figures show a very similar type of distribution. The effect of the coal strike is clearly seen from the records of the automatic filter. The graph for maximum suspended impurity for each day shows a steady fall from the end of April till at the end of September it begins to rise and fluctuate as usual.

ISOLATION AND EXAMINATION OF ATMOSPHERIC DUST BY THE JET APPARATUS.—This instrument (*cf.* ANALYST, 1922, **47**, 322), which was referred to in the 7th Report under the heading "November Fogs," has been further developed; it depends upon the fact that when air containing dust and a sufficiency of water vapour has its pressure suddenly reduced, condensation of the water takes place. This is brought about on a glass surface, and the moisture evaporated, leaving the adhering particles for examination. The whole apparatus is simple and can be purchased at the low price of £4 10s. 0d. The number of particles of dust per c.c. can be determined by means of this instrument; for example, during the dense fog of January 22nd, 1922, a record of 50 c.c. of air taken gave 21,750 particles per c.c., many having a diameter of 1·7 microns, and the average diameter being 0·85 microns. Expired air was also examined for dust particles by this instrument, and the method appears more sensitive than Tyndall's beam of light method. There are other indications of its usefulness in many directions, and work begun with it in connection with acidity and alkalinity has already shown promise.

Work on *Obscurity and Visibility* has not progressed very far during the year owing to difficulties of laboratory accommodation, but measurements on a uniformly illuminated area are expected to begin shortly.

D. G. H.

Fuel Research Board.

PHYSICAL AND CHEMICAL SURVEY OF THE NATIONAL COAL RESOURCES.

REPORT ON METHODS OF ANALYSIS OF COAL.*

THIS report describes in detail the proximate and ultimate analysis of coal, the estimation of phosphorus, caking-index, calorific power, carbon dioxide content, and gives a method for its high and low temperature assay. The important question of sampling is left over for a future report.

The following determinations are carried out on the air-dry sample ground to pass a No. 60 I.M.M. sieve. Moisture is estimated by heating 1 to 2 grms. in flat dishes in an oven at 105° to 110° C., and the ash is obtained by ignition in a muffle attaining a temperature of 750° to 800° C. For volatile matter 1 grm. is heated in a platinum crucible, having a diameter at the base of 24 to 25 mm., and height 35 to 40 mm., and provided with a well-fitting lid, to a temperature of $925° \pm 25°$ C. for seven minutes. A muffle or an electric tube furnace is recommended, but an ordinary open flame may be used if it gives the required temperature, in which case the crucible is supported inside an asbestos-lined conical shield. In the ultimate analysis carbon and hydrogen are estimated in the ordinary way, with the use of lead and a silver spiral; carbon dioxide in the ash should be estimated and allowed for. Sulphur is estimated by ignition with magnesia (or lime), and sodium carbonate in the well known way, and for nitrogen the Kjeldahl method is recommended with the use of mercury or copper sulphate as accelerators, but it is admitted that such results are low. Phosphorus in the ash is precipitated by ammonium molybdate after treatment with *aqua regia*, evaporation of the solution to dryness, and fusion of the residue with sodium carbonate; the precipitate is either dried and weighed or treated with 0·1 N sodium hydroxide solution, the excess of which is titrated with 0·1 N nitric acid.

A modification of the Campredon test is used for determining the caking index. Twenty-five grms. of a mixture of coal and sand (which passes a No. 40 sieve and is retained by a No. 50) are heated exactly as in the estimation of volatile matter; the maximum proportion of sand which allows the coke to support a 500 grm. weight gives the caking index, but the proportion of loose powder in the crucible must not exceed 5 per cent.

In determining the calorific value, which is obtained by combustion in a bomb calorimeter, the usual precautions and corrections are detailed, including the correction for the acids formed.

A laboratory apparatus for the examination of coals by carbonisation at about 600° C. is described, which shows, with fair accuracy, the coke, gas, tar, liquor and ammonia yield which may be expected on the large scale. There are also tables showing the yields obtained from typical coals. For the quick determination of the carbonising characteristics Lessing's coking test is given (*cf.* ANALYST, **1912, 37,** 383), with the recommendation that it be used at 600° and 900° C. for comparative purposes.

A standardised form of report on a coal analysis is added, and an appendix quoting Sinnatt and Harrison's method for the estimation of carbon dioxide (*Lanc. and Cheshire Coal Res. Assoc., Bull.*, 7, 1920). H. E. C.

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* Report No. 2 H.M. Stationery Office, London. 1923. Price 1s. 6d. net.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Analysis of the Jerusalem Artichoke. A. T. Shohl. (*J. Amer. Chem. Soc.*, 1923, **45**, 2754–2756.)—The following percentage composition of the edible portion of Jerusalem artichoke, *Helianthus tuberosus*, found by the author, agrees closely with the results of Strauss (*Berl. klin. Wochschr.*, 1912, **49**, 1213) and Langworthy (*U.S. Dept. Agr. Bull.*, 1917, **468**):—Moisture, 79·0; protein ($N \times 6{\cdot}25$), 3·1; true protein, 0·9; fat, 0·2; carbohydrate, 15·5; fibre, 0·8; and ash, 1·1 per cent. The carbohydrate was estimated by extracting the fresh vegetable or the air-dried material for 72 hours with boiling water. The extract was hydrolysed by refluxing for 2 hours with 10 per cent. of hydrochloric acid and the lævulose was estimated by Benedict's method (*J. Amer. Med. Assoc.*, 1911, **57**, 1193). The carbohydrate (inulin) content was 15·5 per cent. Of the total nitrogen, 71·5 per cent. was water-soluble, and hence not protein. Only small amounts of water-soluble vitamin *B* were present. Analysis, clinical experience and a single experiment in metabolism indicate that the Jerusalem artichoke is a valuable adjunct in the dietetic treatment of diabetes, and its use in this should be re-investigated.

P. H. P.

Estimation of Lipoids and Lipoid-Phosphoric Acid in Flours, Alimentary Pastes, and Eggs. R. Hertwig. (*J. Assoc. Off. Agr. Chem.*, 1923, 7, 91–98.)—The term lipoids denotes the alcohol-ether extract and includes neutral fats, phosphatides, phytosterol, pigments, waxes and cerebrosides. To estimate lipoids in flour, etc., 10 grms. of the finely ground sample are mixed with 30 c.c. of 70 per cent. alcohol and heated at 75° C. for fifteen minutes; 55 c.c. of 95 per cent. alcohol are added, the mixture is shaken for two minutes, then treated with 75 c.c. of ether (dried over sodium), the shaking continued for five minutes, after which the mixture is submitted to centrifugal action and the clear liquid decanted. The residue is extracted with three successive portions of ether saturated with water, and the extracts are added to the alcohol-ether solution. The combined extracts are evaporated, and the residue thus obtained is dried at 100° C. for forty-five minutes and weighed. The dry, weighed residue is then dissolved in 15 c.c. of chloroform, the solution filtered through a small asbestos filter, the filter is washed with chloroform, the filtrate evaporated and the residue dried and weighed. The lipoids thus obtained are boiled with 10 c.c. of alcoholic potassium hydroxide solution, the solution is evaporated, the residue ignited at a low temperature, and the phosphoric acid estimated in the ash. In the case of dried eggs, 3 grms. of the powder are placed on a small asbestos filter and extracted with ether; the extracted powder is then ground with 3 grms. of calcium carbonate, and the mixture is extracted with alcohol-ether, as described for flour. The extracts obtained are added to the original ether extract, which is then evaporated, and the residue of lipoids is weighed, treated with chloroform, etc. To apply the method to liquid eggs, 10 grms. of the sample are shaken with 100 c.c. of ether and 25 c.c. of 95 per cent. alcohol are added gradually; the mixture is submitted to centrifugal action, the solution decanted, and the residue heated at 75° C. for fifteen minutes with 25 c.c. of 70 per cent. alcohol. Forty-six c.c. of 95 per cent. alcohol are then added, and the mixture shaken for two minutes and for a further five minutes after the addition of 70 c.c. of dry ether. From this point the procedure is the same as that for dried eggs. Flour contains from 2·07 to 2·13 per cent. of lipoids and 0·461 to 0·531 per cent. of lipoid P_2O_5; fresh whole eggs 49·7 per cent. of lipoids and 1·299 per cent. of lipoid P_2O_5 (calculated on the dry substance).

W. P. S.

Composition of Pili Nut Oil. A. P. West and S. Balce. (*Philippine J. Sci.*, **1923, 23,** 269–276.)—A sample of pili nuts, *Canarium ovatum*, was purchased in the Manila market, the kernels removed, ground into a meal, and pressed, and after a small amount of stearin had separated from the pale yellow edible oil so obtained its constants were determined as follows:—Sp. gr., 30°/4°, 0·9069; n_D^{30}, 1·4646; iodine value (Hübl), 55·9; saponification value, 197·4; acid value, 1·42; and unsaponifiable matter, 0·19 per cent. A study of the saturated and unsaturated acids led the authors to deduce the composition of the oil to be:—Oleic glyceride, 59·6; palmitic glyceride, 38·2; stearic glyceride, 1·8; and unsaponifiable matter, 0·2; making a total of 99·8 per cent.

D. G. H.

Effect of Composition on the Complete Hydrogenation of Philippine Lumbang and Pili Nut Oils. A. P. West and L. Gonzaga. (*Philippine J. Sci.*, **1923, 23,** 277–292.)—LUMBANG OIL (*Aleurites moluccana*). Hydrogenation with a 3 per cent. of nickel containing catalyst for 20 hours at practically atmospheric pressure gave a fat of melting point 67·5–71·5° C. and iodine value (Hübl) 1·08. Since the oil consists almost entirely of a mixture of linolenic (6·56), linolic (33·48), and oleic (56·98 per cent.) glycerides with 2·85 per cent. of glycerides of solid acids, the theoretical melting point should approximate 71·6° C. (as these glycerides hydrogenate to stearin), and the iodine value be reduced to 0. PILI NUT OIL (*Canarium ovatum*). The composition of this oil (see preceding abstract) is confirmed by the results of hydrogenation. After 15 hours with 3 per cent. nickel catalyst the iodine value (Hübl) was reduced to 0·96, and the melting point was 66–66·8° C. The slightly low melting point may be due to the stearin tending to dissolve in the palmitin, and the melting point of a mixture of stearin and palmitin in the same proportion in which they are found in hydrogenated pili nut oil had a melting point of 68–69° C. Hydrogenation indicates that no saturated glycerides of low melting point are present in this oil.

D. G. H.

Composition of Cashew Nut Oil. A. P. West and C. C. Cruz. (*Philippine J. Sci.*, 1923, **23**, 337–344.)—The cashew nut tree (*Anacardium occidentale*) is widely distributed in the Philippines, and the fruit kernels yield an edible oil resembling olive oil, and a substance known as "cardol" is obtained from the shells. The oil used in the investigation was obtained by pressing the kernels from seeds purchased in the Manila markets, and had the following constants:—Sp. gr., 26·6°/4° C., 0·9105; n_D^{30}, 1·4665; iodine value (Hübl), 85·2; saponification value, 187·0; acid value, 1·45; and unsaponifiable matter, 1·47 per cent., with an iodine value of 94·55. The composition of the residual cake was found to be: Oil, 16·12; moisture, 2·37; ash, 3·94; protein, 31·67; crude fibre, 0·44; and carbohydrates, 45·46 per cent. An examination of the saturated and unsaturated fatty acids indicated that the oil was composed of:—Oleic glyceride, 80·4; stearic glyceride, 17·3; and unsaponifiable matter, 1·5 per cent., making a total of 99·2 per cent.

D. G. H.

Anthocyans in Grapes. R. J. Anderson. (*J. Biol. Chem.*, 1923, **57**, 795–813.)—The author has isolated the pigments occurring in Norton and Concord grapes, and finds them, after purification, to be identical in composition and properties. The colouring matter consists principally of a monoglucoside, anthocyanin, which is similar to oenin derived from *Vitis vinifera*, and only differs in that it gives an intense colour reaction with ferric chloride when dissolved in alcohol. This is similar to that described by Willstätter and Zollinger for the anthocyanin obtained from *Vitis riparia*. The composition of the anthocyanin chloride corresponds to the formula $C_{23}H_{25}O_{12}Cl$. On hydrolysis with boiling hydrochloric acid it yields 1 molecule of glucose and 1 molecule of the sugar-free pigment, anthocyanidin chloride, $C_{17}H_{15}O_7Cl$. The spectrum of anthocyanin chloride consists of one broad band with indefinite margins extending from the yellow into the blue. The anthocyanidin chloride crystallises in beautiful prisms. It is very similar to oenidin chloride, which is obtained on hydrolysing oenin, but it differs in that it contains a lower percentage of methoxyl groups, *viz.* one instead of two. The anthocyanidin chloride, obtained on hydrolysing the glucoside from Concord grapes, appeared to crystallise less readily than the corresponding preparation from Norton grapes, but there was no difference in composition.

P. H. P.

Indian *Artemisia* as a Source of Santonin. (*Bull. Imp. Inst.*, 1923, **21**, 316–318.)—An examination of samples of leaves, flower buds, etc., of *Artemisia brevifolia* showed that in collections made from July 8th, to August 7th, 1922, at Garez, Kashmir, the proportion of santonin increased with the growth of the plant. The proportion of santonin in material received earlier in the year was 0·83 per cent., or 0·92 per cent. on the dry material, but the figures for the later samples were as follows, the first figure in each case representing the percentage of santonin on the wet material, and the second the figure on the dry material:—(1) Leaves and flower buds, July 8th, 0·82 and 0·92. (2) Leaves and flower buds (July 16th), 1·21 and 1·31. (3) Flower buds (July 26th), 0·97 and 1·07. (4) Flower buds (August 7th), 1·64 and 1·79 per cent. Sample (4) was found, on separation, to contain (*a*) in the flower buds and leaves (83 per cent. of sample) 1·95 per cent., and (*b*) in the stalks 1·19 per cent. of santonin. These figures show that *Artemisia brevifolia* compares favourably with *Artemisia maritima*, the usual source of santonin. D. G. H.

Microchemical Identification of Cantharidin. G. Denigès. (*Bull. Soc. Pharm., Bordeaux*, 1923–2; *Ann. Chim. anal.*, 1923, **5**, 332–333.)—About one-tenth mgrm. of the cantharidin is crushed on a micro-slide and dissolved by the addition of a drop of chloroform; the solution is allowed to evaporate, and the resulting crystals are examined without a cover slip; they are rectangular plates arranged like the steps of a ladder. If the crystals thus obtained are not well-defined, a drop of crystallisable benzene is added and allowed to evaporate. In order to confirm the result another slide is placed just over the first one, the area above the crystals is cooled by a drop of water, and the cantharidin sublimed on to the second slide by gentle heat; this produces other characteristic forms of crystals (which may be compared with those from a known cantharidin. Illustrations are given in the original. In toxicological procedure the alkaloidal solution in benzene is evaporated in the above manner, when as little as a few thousandths of a mgrm. may be detected. H. E. C.

Preparation of Commercial Glycyrrhizin. P. Bertolo. (*Giorn. Chim. Ind. Applic.*, 1923, **5**, 497–498.)—Commercial or ammoniacal glycyrrhizin consists of the normal ammonium salt of glycyrrhizic acid, usually mixed with amorphous bitter glycyrrhizin, $C_{35}H_{57}O_{13}N$, and with dark resins soluble in alcohol, yielding an alkaline solution. None of the samples examined by the author exhibited either the desired ready solubility in water to form stable solutions or freedom from appreciable bitterness. The requisite purity and solubility are attained if the liquid extract of liquorice root is concentrated to 12°Be., and the glycyrrhizic acid precipitated by treating each litre of the liquid at a temperature not exceeding 30° C., with 20 grms. of hydrochloric acid diluted with twice its volume of water. The conversion of the glycyrrhizic acid into ammonium salt is then carried out in the ordinary manner. T. H. P.

***Cannabis indica* in Smoking Tobacco. H. Henstock.** (*Pharm. J.*, 1923, **57**, 525.)—*Cannabis indica* was identified in a sample of tobacco as follows:—After drying at 50° C., weighed portions were extracted with (1) *Absolute alcohol*, which yielded a brilliant yellowish-green solution, and a residue which gave a red colour with anhydrous alcohol and hydrochloric acid solution, and when dissolved in acetic acid and treated with concentrated nitric acid, boiled, cooled and diluted with water, gave a dull orange-red precipitate which was taken to be (according to Czerkis, *Ann.*, 1907, 351, 467) the trinitro-carboxylic acid compound of cannabinol, a constituent of *Cannabis indica*. (2) *Light petroleum* which gave a residue yielding on treatment with a few drops of concentrated nitric acid a smell of butyric acid, indicating the presence of cannabinol. (3) *Ether.* The extract, though greenish yellow in colour, was greener than that from other tobacco samples, but not so green as a pure extract of *Cannabis indica.* Further, a quantitative estimation was made by extracting a weighed quantity of the tobacco with absolute alcohol, evaporating the extract to dryness, extracting the residue with ether, and matching the colour of the solution with those of similar solutions obtained in the same way with another tobacco to which known quantities of B.P. *Cannabis indica* had been added. D. G. H.

Detection and Colorimetric Estimation of Hydrocyanic Acid. J. M. Kolthoff. (*Zeitsch. anal. Chem.,* 1923, **63**, 188–190.)—From 5 to 10 c.c. of the solution to be tested are treated with 1 c.c. of a 1 per cent. solution of sodium tetrathionate and 5 drops of 10 per cent. ammonia, and heated in a water-bath at 50° to 55° C. for 5 minutes. To the cooled solution are added 2 c.c. of 4 *N* nitric acid and 3 drops of *N* ferric chloride solution. The sensitiveness of the reaction is 0·0003 grm. of hydrogen cyanide per litre. The conversion into thiocyanate is quantitative; hence the process is suitable for the colorimetric estimation of very small quantities of cyanide: $Na_2S_4O_6 + NaCN + 2NaOH = NaCNS + Na_2S_2O_3 + Na_2SO_4 + H_2O$. The tetrathionate is prepared from equivalent quantities of iodine dissolved in alcohol, and sodium thiosulphate in strong aqueous solution; the crystals are filtered off by suction and washed with dilute alcohol. W. R. S.

Possible Discordant Factor in the Standardisation of Disinfectants. Walker and Weiss. (*The Medical Officer*, May, 1923, Reprint.)—In methods of standardisation which take phenol as the standard, such as the Rideal-Walker test, it is obviously important that the phenol should be pure. The authors point out that, when cresols are present in the phenol, bromine titration is not sufficiently delicate to detect these, and that the test employed should be based on the determination of the solidifying point, with the use of 50 c.c. of the sample. A phenol prepared synthetically solidifies at 40·5° C., whereas one containing 3·4 per cent. of cresols, whatever the particular isomer present, solidifies at 38·8° C. Cresol has three times the bactericidal efficiency of phenol; consequently it will depress the coefficient of a phenol containing it. The authors suggest that no phenol having a solidifying point below 40° C. should be used. Bromine titration should only be used as a check on the gravimetric preparation of the 5 per cent. solution. R. F. I.

Biochemical, Bacteriological, etc.

Copper as a Constituent of Milk. A. F. Hess, G. C. Supplee and B. Bellis. (*J. Biol. Chem.*, 1923, **57**, 725–729.)—The authors describe their investigations on the presence in milk of small amounts of copper by means of the ethyl xanthate method recently employed for this purpose by Supplee and Bellis (*J. Dairy Sci.*, 1922, **5**, 455). The method is based on the fact that small amounts of copper react with potassium ethyl xanthate to produce a yellow colour, which colour is in direct ratio to the amount of copper present. A table shows the amounts of copper recovered from a sample of milk to which definite additions of a copper salt had been made and indicates that the method is reliable for as little as 0·005 mgrm. of copper in 100 c.c. of milk; investigations by the authors showed copper always to be present in larger amounts. A sample of raw cow's milk was found to contain 0·55 mgrm. of copper per litre, and samples of commercially pasteurised milk contained 0·6, 0·7 and 0·6 mgrm. respectively. From analyses, the copper content of human milk is judged to be about the same as that of the milk of the cow. These results make it appear probable that copper has some physiological action. P. H. P.

Quantitative Study of the Destruction of Vitamin *B* by Heat. H. C. Sherman and M. R. Grose. (*J. Amer. Chem. Soc.*, 1923, **45**, 2728–2738).—The authors describe the effect of 4 hours' heating upon vitamin *B* of tomato juice at 10° C. intervals over the range of 100° to 130° C. They employed the rat-growth method as a measure of vitamin *B* content. At 100° C., in acid medium, destruction occurred owing to the hot water. The following are the average figures for the destruction of vitamin *B* in tomato juice for a period of 4 hours' heating, derived by determining the size of heated doses at each temperature necessary to give weight curves approximately coinciding with those of positive controls fed with 4 c.c. of unheated juice:—At 100° C., 20 per cent., at 110° C.; 33 per cent.; at 120° C., 47 per cent.; and at 130° C., 55 per cent. A mathematical computation bringing the comparison curves into exact coincidence gives the following:—At 100° C., 24 per cent.; at 110° C., 33 per cent.; at 120° C., 45 per cent.; and at 130° C., 58 per cent. Vitamin *B*, therefore, like vitamin *C*, has a low temperature coefficient of heat destruction, only 1·3 to 1·4 at this range, as compared with 2 in most chemical reactions. The heat destruction of the vitamin, in marked contrast with the heat coagulation of typical proteins, and with the heat destruction of such typical enzymes as have been investigated, showed no increase in the temperature coefficient at temperatures in the neighbourhood of 120° C. It followed the orderly course of a chemical reaction under the accelerating influence of heat, but with a less than average temperature coefficient. P. H. P.

Investigation and Application of the Rat-growth Method for Studying Vitamin *B*. H. C. Sherman and A. Spohn. (*J. Amer. Chem. Soc.*, 1923, **45**, 2719–2728.)—The selection and care of the animals used and the basal diets given are described. Eight c.c. of fluid skimmed milk or 0·8 grm. of dry milk per rat per day, when fed separately on the basal ration, were the quantities chosen as being most advantageous to give in order to detect possible reduction in vitamin *B* content on heating. It is believed to be possible to detect a diminution, certainly of 25 per cent., and probably of 15 per cent., in the vitamin *B* content of the food tested when dealing with averages of 10 or more suitably chosen rats on each diet. Vitamin *B* in the form in which it exists in milk is comparatively stable to heating at 100° C. in the dry state, no diminution being noticed after 48 hours of heating with free access of air, but it is less stable when heated at 100° C. in aqueous solution. After 6 hours there was an apparent diminution; probably a fourth of the vitamin was thus destroyed. P. H. P.

Two Methods for Studying Vitamin *B*. H. C. Sherman and H. Edgeworth. (*J. Amer. Chem. Soc.*, 1923, **45**, 2712–2718.)—The authors have examined the availability of the gravimetric yeast-growth method and of the rat-growth method for the quantitative study of vitamin *B* and their experiments are described. Both methods yield quantitative results. The probable errors of the averages obtained in the different series by the former method are of the order of 1 per cent., and by the latter method of 2 to 4 per cent., of their numerical values. The increased growth of yeast may, however, be due to the introduction of other substances favourable to yeast growth. The rat-growth method is therefore considered preferable, in spite of the larger probable errors, since the results can be interpreted in terms of vitamin *B* with much greater certainty.

P. H. P.

Toxicological and Forensic.

Solubility of Mercuric Sulphide in Ammonia and its Influence on the Detection of Arsenic in the Presence of Mercury. C. Ghigliotto. (*Ann. Chim. anal.*, 1923, **5**, 326.)—The precipitate which always forms on passing hydrogen sulphide through the solution obtained in the destruction of organic matter by the wet method commonly used in toxicological analysis invariably contains organic matter, as well as mercury sulphide, if any. If this precipitate is treated, as usual, with ammonia the mercury is partially dissolved, and this may give rise to two errors; the presence of mercury may be overlooked if the precipitate entirely dissolves, or the presence of mercury in the filtrate may prevent the formation of the mirror due to arsenic when the solution is treated by the Marsh process. H. E. C.

Composition and Toxicity of the Arsenobenzenes. Estimation of Arsenic in these Products. M. de Myttenaere. (*Bull. de l'Acad. Roy. Med. Belg.*, 1923, **3**, 258; *J. Pharm. Chim.*, 1923, **28**, 357–362.)—The method of estimating the arsenic is based upon its oxidation by means of 10 vol. hydrogen peroxide. The sample (0·2 grm.) is placed in a 200 c.c. conical flask and dissolved in 5 c.c. of water. Ten c.c. of hydrogen peroxide are added, followed by 10 c.c. of 50 per cent. (by weight) sulphuric acid, and the mixture gently boiled on a sand bath. When decomposition of the hydrogen peroxide is complete, the liquid is cooled, a further 5 c.c. of sulphuric acid added, and the whole re-heated until colourless and white fumes of sulphuric acid are formed. Ten c.c. of water are then carefully added, and a few drops of 1 per cent. potassium permanganate solution till the colour persists. Decolorisation is then effected by means of 2 per cent. oxalic acid, 20 c.c. of 25 per cent. potassium iodide solution are added, and the liquid heated for 20 minutes on a water bath, cooled, and exactly decolorised with

0·1 *N* sodium thiosulphate. Twenty-five c.c. of a cold saturated solution of sodium carbonate are next added, and an excess of sodium bicarbonate, and the arsenic in the original 0·2 grm. estimated by titrating the solution with 0·1 *N* iodine solution, and multiplying the number of c.c. required by 0·003748.

In considering the formula of the arsenobenzenes one notes that there are 2 arsenic and 2 nitrogen groups in the central nucleus, whatever the composition and complexity of the side chains—$\left(\frac{\text{As}}{\text{N}} = \frac{75}{14} = 5{\cdot}357\right)$. Thus a product containing arsenic and nitrogen in the theoretical proportions should give figures for arsenic and nitrogen as 5·357 is to 1. Toxic products are shown to have a deficiency of nitrogen in proportion to arsenic. Further, in such toxic products the amount of arsenic precipitated by hydrogen sulphide (DM1) increases in proportion to the toxicity; it is estimated as follows:—One grm. of the product is dissolved in 90 c.c. of boiling distilled water, 10 c.c. of 30 per cent. acetic acid added, and the whole heated in a steam bath (with stirring from time to time) for 10 minutes from the time precipitation begins. After cooling and filtering, the clear filtrate is heated to about 60° C., 5 c.c. of hydrochloric acid added, and hydrogen sulphide passed in to saturation. After standing and filtration, the precipitate is washed with warm water, digested with 20 c.c. of ammonium carbonate solution, washed with distilled water, and the ammonium carbonate boiled off on a water bath. Ten c.c. of hydrogen peroxide and 10 c.c. of 50 per cent. sulphuric acid are carefully added, and the liquid evaporated till white fumes appear. A few drops of 1 per cent. potassium permanganate solution are added, and the operation continued as above. The number of c.c. used in the titration (DM1) should not exceed an outside limit of 12, but in the course of the investigation some ampoules of the same series were found to give varying results, probably owing to faulty vacuum and oxidation, and details of a study of this point are given. Particulars are also given for the estimation of the additional amount of arsenic found in the various precipitates encountered in the investigation, and the composition of these precipitates is discussed. The general conclusion drawn is that a product which may be regarded as satisfactory from the toxicological point of view should contain a total amount of arsenic between 19 and 21 per cent., that the ratio of arsenic to nitrogen should not exceed 5–6, and that (DM1) must not exceed 12. D. G. H.

Toxicological Detection of Minute Quantities of Nitric Acid. C. Ghigliotto. (*Ann. Chim. anal.*, 1923, **5**, 325.)—The membranes and muscles of the stomach act as a mordant in combining with nitric acid, even in very dilute solutions, as does the skin when it forms the familiar yellow stain with the strong acid. This forms the basis of the following method for the detection of the acid in the stomach in quantities far too small to be detected by the ordinary process of distillation. Pieces of the membrane are washed with water, dried between filter paper, and a few drops of diphenylamine in sulphuric acid are added; the characteristic blue colour is developed, even with the acid so dilute as 1:1000.

H. E. C.

Water Analysis.

Estimation of Dissolved Oxygen in the Presence of Iron Salts. A. M. Buswell and W. U. Gallager. (*J. Ind. Eng. Chem.*, 1923, **15**, 1186–1188.)—The Rideal and Stewart modification of the Winkler method for the estimation of dissolved oxygen in waters yields high results when the water contains iron salts; the presence of 1 part per million of iron causes the result for the oxygen to be from 0·1 to 0·13 part per million too high, but a satisfactory correction cannot be applied, since, according to circumstances, from 75 to 90 per cent. of the iron reacts. Trustworthy results may, however, be obtained by Mohr's method as modified by Levy, in which ferrous iron in alkaline solution is oxidised by the dissolved oxygen. A portion of the water is treated with standard ferrous iron solution, then rendered alkaline with potassium hydroxide solution and acidified; another similar portion of the water is acidified, and then treated with the ferrous iron solution. Both portions are titrated with permanganate solution and the difference between the two titrations is proportional to the amount of dissolved oxygen present. The apparatus used consists of a bulb with a tap at each end and a funnel at one end for the introduction of the reagents. W. P. S.

Agricultural Analysis.

Estimation of the Sulphur Compounds in Dry Lime Sulphur. C. P. Jones. (*J. Agric. Res.*, 1923, **25**, 323–336.)—When carbon dioxide is passed through a solution of polysulphide the monosulphide sulphur is completely separated from the thiosulphate and residual sulphur; this fact forms the basis of the following method for the estimation of the three forms of sulphur in dry lime-sulphur. About 0·5 grm. of the sample is placed in a dry flask fitted with inlet and outlet tubes and a dropping funnel. The inlet tube delivers a stream of carbon dioxide which has been passed through sodium bicarbonate solution (to remove traces of free acid) and through sulphuric acid. The outlet tubes pass into two wash bottles which contain 100 c.c. of hydrated sodium peroxide solution. After all air has been displaced by the carbon dioxide 50 c.c. of water are run into the flask, which is then shaken for five minutes without interrupting the current of gas, and the gas passed until all the hydrogen sulphide has been expelled and absorbed by the alkaline solution. The contents of the flask are filtered through asbestos, and the filtrate diluted and titrated with 0·05 *N* iodine solution to estimate the thiosulphate. The sulphur on the filter and any adhering to the tubing is washed with dilute hydrochloric acid, dried at 100° C., weighed, ignited, and again weighed; the difference gives polysulphide sulphur together with any free sulphur. The sulphur evolved as hydrogen sulphide is oxidised to sulphate by the peroxide solution, which is then boiled and the sulphate precipitated with barium chloride. H. E. C.

Estimation of Nitrate Nitrogen in the Presence of Cyanamide and some of its Derivatives. K. D. Jacob. (*J. Ind. Eng. Chem.*, 1923, **15**, 1175–1177.)—An aliquot portion of the solution containing nitrate, urea, cyanamide, dicyanodiamide, etc., is neutralised and treated with 10 c.c. of a neutral 2 per cent. extract of jack-bean flour; after one hour the urea present will have been converted into ammonia and carbon dioxide. The mixture is then treated with 100 c.c. of saturated silver sulphate solution and 10 c.c. of 15 per cent. potassium hydroxide solution, and filtered after one hour, and the precipitate is washed six times with 10 c.c. portions of water. This treatment removes the cyanamide, dicyanodiamide and guanyl-urea. The filtrate is diluted to 350 c.c., treated with 5 c.c. of 20 per cent. sodium hydroxide solution and distilled to remove ammonia; 300 c.c. of distillate are collected. Two hundred c.c. of water and 2 grms. of Devada's alloy are then added to the residual solution in the distillation flask, and the distillation is continued, 200 c.c. of distillate being collected in a definite volume of 0·1 *N* sulphuric acid contained in the receiver. The excess of acid is titrated subsequently, and the ammonia thus found represents the nitrate present in the original solution. W. P. S.

Organic Analysis.

Estimation of Phosphorus in Organic Substances. J. Garola. (*Ann. Chim. anal.*, 1923, **5**, 326–328.)—The ordinary incineration of organic matter leads to the loss of phosphorus, and the destruction of large quantities of such material by the Kjeldahl process is tedious. Accurate results may be obtained by ignition in the presence of calcined magnesia. To about 5 grms. of the mixture to be examined is added about 0·2 grm. of magnesia, which is intimately mixed in by making a paste of the mixture with water and drying on the water bath prior to the ignition. The ash is extracted with dilute hydrochloric acid, the solution filtered and diluted to about 100 c.c., and the phosphate precipitated as ammonium phosphomolybdate. H. E. C.

Detection of Esters of Fixed Acids in Essential Oils. A New Method of Testing. C. T. Bennett and D. C. Garratt. (*Perfumery and Ess. Oil Record.*, 1923, **14**, 359.)—As little as one per cent. of ethyl citrate, tartrate, succinate, benzoate, phthalate or cinnamate may be detected in essential oils by the following simple procedure:—One c.c. of the essential oil and 3 c.c. of an approximately 10 per cent. solution of potassium hydroxide in absolute alcohol are placed in a test tube, and the whole heated in a water bath for a few minutes and cooled. The oil may be regarded as unadulterated with the above esters if no precipitate is formed in, at most, an hour. Ethyl phthalate (2·5 and 1·0 per cent.) very quickly causes a precipitate; with 2·5 per cent. of the cinnamate a precipitate forms rapidly, and with 1 per cent. more slowly; with 2·5 per cent. of the succinate a gelatinous mass results, and with 1 per cent. a crystalline precipitate; citrate and tartrate cause cloudiness and crystals are precipitated on standing; with 2·5 per cent. of benzoate precipitation is slow. The test appears to be specially delicate for phthalic esters. D. G. H.

Estimation of Pentoses and Pentosans. 1. The Formation and Distillation of Furfuraldehyde. N. C. Pervier and R. A. Gortner. (*J. Ind. Eng. Chem.*, 1923, **15**, 1167–1169.)—The usual method of estimating pentoses consists in distilling the substances with 200 c.c. of 12 per cent. hydrochloric acid at such a rate that 30 c.c. of distillate are collected in ten minutes; 30 c.c. more of the 12 per cent. acid are then added to the distillation flask and the process is repeated until 360 c.c. of distillate have been collected. The resulting furfuraldehyde is estimated in the distillate. It is shown that during this process of distillation the concentration of the acid varies from 12 to 20 per cent. and that the stronger acid has a destructive effect on the furfuraldehyde, thus causing the method to yield low results. The following method of distillation prevents loss of the aldehyde:—A quantity of 0·2 to 5·0 grms. of the substance (according to the pentose or pentosan content) is placed in a distillation flask, together with 200 c.c. of 12 per cent. hydrochloric acid (sp. gr. 1·06), and a slow current of steam is led into the mixture; as soon as the liquid boils the flask is heated by means of a small burner so that the temperature of the vapours is maintained between 103° and 105° C., as measured by a thermometer in the neck of the flask. The steam distillation is continued until a drop of the distillate no longer gives a red coloration after three minutes' contact with a drop of aniline acetate solution on a filter paper. Theoretical yields of furfuraldehyde were obtained when this method was applied to *l*-arabinose and *d*-xylose. W. P. S.

Quantitative Variation of Gossypol and its relation to the Oil Content of Cottonseed. E. W. Schwartze and C. L. Alsberg. (*J. Agric. Res.*, 1923, **25**, 285–295.)—Examination of many varieties of cottonseed has proved the presence of gossypol in the kernels of all varieties and the husks of some. The amount is very variable, being from 0·4 to 1·2 per cent. even in different samples of the same variety on the same plantation in different years, but is proportional to the oil content. Estimation of the gossypol is carried out by a modification of Carruth's aniline method (ANALYST, 1918, **43**, 222.) Seventy-five grms. of the sample are thoroughly extracted in a Soxhlet tube with ether, which is then distilled off and the extract dissolved in 8 volumes of petroleum spirit. After standing overnight the flocculent precipitate, which usually appears, is filtered off and washed with ether, the washings evaporated, and the residue dissolved in petroleum spirit and added to the main solution. One c.c. of aniline is added, and the mixture shaken for 5 minutes, or until all the aniline is dissolved, then set aside for 3 to 7 days, after which the aniline-gossypol is filtered on to a Gooch crucible, washed with petroleum spirit, dried at 100° C. and weighed. The weight of the precipitate, less the weight of the aniline in it (N × 6·64), gives the weight of the gossypol. The latter may be identified by means of its acetate, the crystallographic details of which are given in the paper. H. E. C.

Chrome-leather Analysis. International Commission Report. R. F. Innes. (*J. Soc. Leather Trades Chem.*, 1923, 413.)—An official method for estimating chromium has been adopted in the leather trade. It consists in fusing in a nickel crucible the ash from 5 grms. of the sample with five times its weight of sodium peroxide for one to five minutes at a moderate red heat. The fused mass is allowed to cool, dissolved in hot water, and boiled for 10 minutes in the presence of a clean piece of thin sheet iron, about half-an-inch square. The solution is filtered, the residue washed, and filtrate and washings diluted to 500 c.c. One hundred c.c. are then acidified with hydrochloric acid free from chlorine, and the chromium estimated iodimetrically.

An alternative and quicker method is also allowed:—The ash from 5 grms. of the sample is mixed and ignited in a platinum crucible with three to four times its weight of a mixture of equal amounts of magnesia and sodium carbonate for one minute. The mixture is carefully and thoroughly ground in an agate mortar till homogeneous and roasted again in the platinum crucible at a bright red heat for ten minutes. The mixture is then dissolved in excess of dilute hydrochloric acid, and the chromium estimated iodimetrically.

A provisional method has been adopted for estimating free sulphur and grease by extracting the sample in a Soxhlet extractor for 3 hours with petroleum spirit. The free sulphur alone is estimated by treating the mixture with fuming nitric acid at laboratory temperature for 3 days, whereby sulphuric acid is formed; this is subsequently estimated gravimetrically. R. F. I.

Effect of Perspiration on Chrome Upper Leather. R. F. Innes. (*J. Soc. Leather Trades Chem.*, 1923, 436.)—A glacé kid shoe-front had worn very badly and become very firm, badly cracked and dull in appearance. This effect had been produced by wear on a hot foot. A strip about ¼ inch wide outside the stitches was cut off, as this showed the bright glaze and had apparently been protected from the action of the perspiration. The two pieces were analysed, and showed the following results:

	Cracked.	Unaffected.
Chromic oxide, per cent.	1·71	1·90
Sodium chloride, per cent.	2·75	0·69
Behaviour on boiling	much shrivelled	somewhat shrivelled

The low chromium indicates a semi-chrome leather. Perspiration evidently has a deleterious effect on this type of leather. R. F. I.

Inorganic Analysis.

Colorimetric Estimation of Small Quantities of Bismuth. L. Cuny and G. Poirot. (*J. Pharm. Chim.*, 1923, 28, 215–223.)—The method is based upon the fact that if an aqueous solution of quinine sulphate and potassium iodide is added to a solution of a bismuth salt containing sufficient gum arabic, no precipitate of iodobismuthate of quinine is formed, but a yellow colour develops similar to that of potassium dichromate solutions.

After experimenting with different strengths of reagents and conditions of reaction, the following method of procedure was adopted: Solutions required are (1) *Stock solution of bismuth.* Dissolve 2·23 grms. of bismuth oxide in 10 per cent. nitric acid and make up to a litre. (2) *Standard solution of bismuth.* Take 25 c.c. of solution (1), add 175 c.c. of 10 per cent. nitric acid, and make up to a litre with distilled water. Five c.c. of this solution will thus contain 0·25 mgrm. of bismuth and 0·1 mgrm. of nitric acid. (3) *Quinine nitrate.* Dissolve 1 grm. of the hydrate in 5 c.c. of 10 per cent. nitric acid, and make up to 100 c.c. with distilled water. (4) *Five per cent. potassium iodide solution.* (5) *Ten per cent. gum arabic solution in ampoules.* Five c.c. of the standard solution and 5 c.c. of the solution to be examined are respectively measured into dry test tubes, 3 c.c. of the gum arabic and 1 c.c. of the potassium iodide solution added, with shaking after each addition, and the colours compared in a Dubosq colorimeter. The standard solution is at a height of 5 mm., and the height found on matching the second solution should lie between 4 and 7 mm., suitable dilution of one or other of the solutions being effected if necessary. Under these conditions the proportion of bismuth is found from the formula—$x = \frac{50 \times 5}{n}$, where n is the reading obtained. Where bismuth was estimated by this method in solutions of known strength the percentage error in 6 estimations varied from $-0{\cdot}3$ to $+3{\cdot}5$. Errors are introduced by the presence of free hydrochloric, sulphuric and acetic acids, traces of nitrogenous substances capable of decomposing the potassium iodide, and salts of metals, although in the last case the gum arabic tends to prevent the formation of precipitates and to give colloidal solutions. D. G. H.

The Gravimetric Ratio—Antimony : Antimony Tetroxide. J. Knop. (*Zeitsch. anal. Chem.*, 1923, **63**, 181–188.)—Finely powdered antimony of known purity was carefully oxidised with nitric acid and the dry oxide heated for one hour at 850° to 900° C. in an electric furnace, the temperature of which was observed by means of a thermo-couple. The experiments, which were carried out with every precaution to ensure an accuracy of ±0·0001 grm., gave the equivalent Sb = 122·04, or 121·94 after reduction to weight *in vacuo.* The tetroxide obtained was distilled with hydrochloric acid and potassium iodide in an atmosphere of carbon dioxide, and the iodine titrated with thiosulphate solution standardised against dichromate; these determinations gave a mean of 121·98. W. R. S.

Separation of Radium and Barium. V. Chlopine. (*Bull. Soc. Chim.*, 1923, **33**, 1547–1551.)—The method of separating radium and barium elaborated by the author is based on the regular diminution produced in the solubility of barium chloride in water by gradual addition of hydrochloric acid, and on the fact that, when gaseous hydrogen chloride is passed into a solution of barium chloride containing radium, the precipitated chloride is richer in radium than the chloride remaining dissolved. This method is as effectual as fractional crystallisation with the help of evaporation; it also allows of the ready variation of the coefficient of enrichment within wide limits and does not require preliminary purification of the chlorides; the presence of lead is without harmful effect on the coefficient of enrichment. T. H. P.

Identification of Amines. C. S. Marvel and F. E. Smith. (*J. Amer. Chem. Soc.*, 1923, **45**, 2696–2698.)—In the Hinsberg test (*Ber.*, 1890, **23**, 2963), *p*-bromobenzenesulphonyl chloride cannot be used in place of benzenesulphonyl chloride, because, although it gives easily crystallisable *p*-bromobenzenesulphonyl amides with sharp melting points (these being especially good for ethyl aniline, *n*-propylaniline, *n*-butylaniline and piperidine), yet, with many primary amines it gives alkali-insoluble derivatives. The preparation of *p*-bromobenzenesulphonyl chloride is described. The amide is prepared by treating the chloride with a slight excess of the amine and warming, if necessary, to start the reaction, which takes place smoothly with the evolution of heat and is complete in a few minutes. The product is washed with dilute hydrochloric acid, to remove excess of amine, and then recrystallised from alcohol. Methylamine, dimethylamine and piperidine derivatives are prepared by the action of *p*-bromobenzenesulphonyl chloride and aqueous alkali in the usual manner on an aqueous solution of the amine hydrochloride. The following is a list of certain amines with the melting points of the corresponding para-bromobenzenesulphonamides:—Methylamine, 77° C.; benzylamine, 117° C.; dimethylamine, 94° C.; piperidine, 91° C.; *o*-toluidine, 116° C.; *p*-toluidine, 98° C.; *p*-anisidine, 142° C.; *p*-phenetidine, 143° C.; *α*-naphthylamine, 183·5° C.; and *β*-naphthylamine, 129° C. P. H. P.

Hypobromous Acid and Estimation of Hypobromous and Bromic Acids. E. Biilmann and E. Rimbert. (*Bull. Soc. Chim.*, 1923, **33**, 1465–1473.)—For estimating bromic and hypobromous acids together use may be made of the fact that the latter acid reacts instantaneously with excess of phenol, forming brominated derivatives which have no action on potassium iodide, whereas bromic acid does not react with phenol, but liberates iodine from the iodide in presence of dilute sulphuric acid, and may thus be estimated by subsequent titration of the solution with thiosulphate. In concentrated solutions the reaction between bromic and hydriodic acid takes place sufficiently rapidly, but the liquid should then be diluted before titration with thiosulphate; when dilute solutions are used, the reaction should be accelerated by means of a catalyst, such as ammonium molybdate (ANALYST, 1921, **48**, 160). Trial analyses on these lines yielded satisfactory results. T. H. P.

Physical Methods, Apparatus, etc.

Method for the Separation of Gas Mixtures. M. Shepherd and F. Porter. (*J. Ind. Eng. Chem.*, 1923, **15**, 1143–1146.)—A method is described for the separation of the constituents of a gas mixture by fractional distillation at low temperatures and pressures. The apparatus used consists of one complete unit combining all the parts necessary for condensation, distillation, pumping, storage, measurement, and distribution. The distillation bulbs are surrounded by Dewar tubes containing liquid air, and these tubes are connected with a vacuum pump or mercury pressure seals, so that the air may be made to boil at pressures varying from 0·1 to 2 atmospheres and at a corresponding temperature of −180° to −208° C. Details of the application of the method to the analysis of a gas mixture containing helium, nitrogen, methane, ethane, propane, butane and higher saturated hydrocarbons are given. W. P. S.

Behaviour of Pumice Stone during the Dehydration of Organic Liquids. A. Seidenberg. (*J. Assoc. Off. Agr. Chem.*, 1923, 7, 99–106.)—Ignited pumice stone readily absorbs moisture from liquids spread over it, and this absorbed moisture can only be removed completely by again heating the pumice to redness. The absorbed moisture has also a decided effect in accelerating the decomposition of organic substances spread over the surface of the pumice, which is, therefore, an unsuitable substance to use in aiding the drying of these liquids. If the temperature used is low, all the moisture is not expelled, and at a higher temperature it is not possible to distinguish between loss due to decomposition and loss due to evaporation of water. W. P. S.

Colour Measurement of Tanning Materials. T. Blackadder. (*J. Soc. Leather Trades Chem.*, 1923, 445.)—This method obviates the faults in the Lovibond method due to partial colour-blindness in different observers, those due to varying illumination, and those due to the optically impure standard glasses. (It is possible to match a chestnut extract with or without blue in the combination at will). Theoretically one can measure coloured light qualitatively by resolving it by means of a spectroscope which shows the absence or preponderance of certain colours or wave-lengths. To measure these quantitatively one should measure the amount of every wave-length, but a practical approximation can be obtained by measuring a large number of the wave-lengths at scattered parts of the spectrum. If one passes a beam of light through a tannin solution one can measure the intensity of as many wave-lengths as is considered practicable and express this in terms of the intensity of the original beam, thus obtaining a specific measure of the colour of the light passed by the solution and also of the amount absorbed by the solution. In this way one is independent of the purity of the original source of light. Trials have shown that four particular regions are most suitable for tannin solutions:—Red, passing wave-lengths of 700–600; yellow, passing wave-lengths of 550–600; green, passing wave-lengths of 510–550; and blue, passing wave-lengths of 400–510. For other solutions, such as dyes or oils, another set of three or four regions might be more suitable.

In practice, use is made of a double field of some design which is evenly illuminated when distilled water is in the position afterwards occupied by the solution. The incident light passes through a variable opening in a rotating sector to the first half of the field and through a cell containing the tannin solution to the second half. A half light is used on the first half by the use of a neutral tint filter of 50 per cent. transmission or a rotating disc, one half of which has been cut away. The thickness of the test solution is varied till a match to this is obtained. Therefore the lighter the colour of the solution, the greater the proportion of the light it will pass and the higher the measurement. This is the reverse of the Lovibond method.

The apparatus used is of the type described by Schmidt and Haensch (*J. Amer. Leather Chem. Assoc.*, 1922, 209). The individual measurements are made by observing the field of the instrument through each of the four colour filters in turn. These with the neutral tint filter can be obtained from the Eastman Kodak Company already standardised. The concentration of the tannin solution is 0·4 per cent., as used in the official analytical method. Readings are given of several tannin extracts by the new method; also a comparison of it with the Lovibond method. Another method of expressing the results is to reduce the red readings to 10·0 and the other colours in proportion. R. F. I.

Note by Abstractor.—Attention may be drawn to Prof. Procter's paper on this subject in *J. Soc. Chem. Ind.*, **42**, 73T–79T; ANALYST, 1923, **48**, 405.

Method of Micro-incineration applicable to Histo-chemical Investigations. A. Policard. (*Bull. Soc. Chim.*, 1923, **33**, 1551–1558.)—Before being sectioned, the tissue or organ is fixed by means of a coagulating agent which neither adds nor removes mineral constituents. Neither acid nor saline fixing agents, such as bichromates, mercuric chloride, etc., should be used. The most satisfactory agents are alcohol and formaldehyde, although the former cannot be used when phosphorus is sought, since it removes phosphorus-containing lipoids. The fixed material is frozen by means of solid carbon dioxide and cut into sections, which are collected in water or alcohol and then spread on slides and dried, away from dust, at first in the air and later in an oven at 58° C.; a final drying at 105° C., prior to incineration, is also advisable. The incineration is best effected by raising the temperature in 12 to 15 minutes to dull redness. For this purpose use may be made of a small electric furnace consisting of a quartz tube surrounded by a resistance and by a suitable asbestos covering. The glass slide is conveniently supported either on unglazed porcelain or on platinum foil and this on asbestos card; inclination of the tube causes a slight draught, which favours the calcination.

The ash is viewed by reflected light under a magnification of 60 or, better, 150 diameters, which renders possible the distinction of many elements, especially of those of the cellular nucleus. A lamp similar to the Stiassnie ultra-microscopy lamp permits a convenient orientation of the light. The fragile ash may be protected by a cover-glass fixed at the corners with paraffin-wax. T. H. P.

Reviews.

LABORATORY MANUAL OF PHYSICAL CHEMISTRY. A. W. DAVIDSON and H. S. VAN KLOOSTER. Pp. 182. New York: John Wiley & Sons; London: Chapman & Hall. 1922. Price 10s. net.

This volume will certainly be useful for the somewhat narrow purpose for which it is intended. It is designed to assist the instructor in handling a large class of beginners, and to enable the students to record the results of a large number of experiments in a minimum of time. This narrow scope obviously restricts the general utility of the book, but, bearing in mind the avowed object, it must be said that the volume presents good points—and some bad ones.

There are described in detail the method of carrying out some twenty-four well-selected experiments covering the range of physical chemistry usually required from university students not reading for honours in this branch. With the possible exception of the Emerson type of bomb calorimeter, all the apparatus described is that common to well-equipped laboratories. For each of the experiments there is given a list of references to theoretical text-books dealing with it, a list of apparatus required, an illustration and blank spaces for recording the results of observations; the book therefore is to serve as a note-book as well as a text-book. The production of the book is excellent in printing, paper and cost. Very few errors were observed, as is to be expected from such experienced authors.

There is a decidedly American style about the volume; although containing an abundance of blank pages and waste space the abbreviations frequently used are a blemish, "vis. and surf. tens." for "viscosity and surface tension" may be allowable in a laboratory note-book, but such expressions do not look well in print. Most chemists, even elementary students, do not need telling for each experiment that a Bunsen burner requires rubber tubing, or that a condenser requires the support of an iron stand; if such a hint is necessary, the many excellent drawings suffice to give it. Had much of the repetition in the lists of books and apparatus been withheld there would have been space for the methods of calculation and a brief statement of the theory underlying the experiments. It appears that the student goes the round fitting up apparatus laid out like "Meccano" parts and reading results. This method seems to detract from the educational value of the work, but doubtless assists in the turning out of chemists, like Ford cars, by the million.

H. E. COX.

SECOND YEAR COLLEGE CHEMISTRY. WILLIAM H. CHAPLIN. Pp. xi.+311. New York: John Wiley & Sons; London: Chapman & Hall. Price 15s. net.

A review of the laboratory companion of this text-book appeared in THE ANALYST for last June, and many of the comments made there apply also to this volume. The title is a misnomer; "Outlines of Physical Chemistry" would convey a more accurate impression of the scope of the work.

In addition to the subjects referred to in the above-mentioned review, this book contains chapters on the Periodic system, Radioactivity and Atomic Structure; the two latter give an up-to-date account of radioactive transformations, and introduce recent work of Rutherford, Langmuir and others on the constitution of atoms. Many of the chapters commence with a useful outline of the historical development of the subject, and the treatment throughout is clear and concise; the illustrations are easy to follow and sufficiently numerous for their purpose; a considerable variety of test questions is also provided.

A particularly satisfactory section is that on indicators, the discussion of this subject being unusually complete, although the P_H notation might, perhaps, have been introduced with advantage. On the whole, this volume leaves a more favourable impression than its laboratory companion, and it can be recommended for the study of elementary physical chemistry. A reduction in price would put this book more on a level with others on the same subject. A. F. KITCHING.

REDWOOD AND EASTLAKE'S PETROLEUM TECHNOLOGIST'S POCKET BOOK. 2nd Edition. Pp. 546. London: Charles Griffin & Co., Ltd. 1923. Price 15s. net.

One can only say of this book that it appears to be a compendium of all the information that a prospector for oil could possibly require—general, statistical, geological, legal, or linguistic. Surface indications, testing for oil, trial borings, purchase or leasing of land (in any country, speaking any language), storage, transport, refining of the product, all are described. To indicate the variety of its contents even in summary would require several times the space that could be allotted to a review in the ANALYST, and those really wishing to know what is in the book must consult the table of contents, itself occupying 16 pages, or the excellent index.

Much of the matter, such as the capacity of wheelbarrows, the force of the wind, the sizes of wire nails, the Roumanian for seventeen, or the distance from Constantinople to Dover, is only of secondary interest to analytical chemists; but the book is a mine of such miscellaneous information, all, of course, bearing on one phase or other of the petroleum industry.

The most useful portions of the book to chemists are the long tables of specific gravities and of the results of fractional distillation of actual individual samples of oils from every part of the world, of the composition of natural gases, asphalt rocks and refined asphalts; and the similar, though smaller, collection of calorific values and specific heats of oils. Matter is brought together here which, without this book, it would be very difficult and tedious to find.

The testing of petroleum and of asphalts is mentioned: but for the most part individual tests are merely enumerated, or the methods of carrying them out indicated in the most general terms. Whilst this is all that is done for such common constants as specific gravity, viscosity, or flash point, or for the fractionation of oils by distillation, yet the method for the quantitative bromination of an oil is quoted at length and in detail from Allen's *Organic Analysis*. It seems to the reviewer that either this process should have been merely indicated, like the others, or that some space should have been devoted to a description of the methods used in determining the various constants mentioned.

There are, of course, the usual tables of weights and measures of all nations, and tables for their conversion into metric equivalents, and the conversion of hydrometer readings and thermometric scales, which make one once more marvel at the perversity of mankind in refusing to rid themselves of the useless and wasteful labour which the existence of such tables implies; and there are some unusual and useful (the pity is that such things should be useful) tables for the mutual conversion of the readings of Redwood, Engler, and Saybolt viscometers. The figures, as far as the reviewer has tested them, seem to have been carefully edited, and errors are rare, though there is rather a bad one in the formula given on page 308 for the capacity in imperial gallons of a barrel or cask. One regrets the misuse of language involved in the use of the term "water white" for colourless; but its use by either a poet or a petroleum prospector does not merit the same reprehension as its issue in a specification for "Benzole" by a professedly scientific body like the Engineering Standards Association. Yet who knows what effect the term may have on the morals of a man who is thinking of adulterating milk? The style of the book is excellent, all the work in it seems to be very well done, and the index makes it all readily accessible. To the petroleum technologist the book must be invaluable, and the chemist will find in it much valuable material that he will not easily find elsewhere. J. T. DUNN.

THE MANUFACTURE OF NITRIC ACID AND NITRATES. By A. COTTRELL, M.Sc., F.I.C. (Lunge and Cumming). Pp. xv. + 454. London: Gurney & Jackson. 1923. Price 36s. net.

This is the third volume to appear of the revised edition of Lunge, which is being edited by Dr. Cumming. This volume deals somewhat exhaustively with nitric acid manufacture, and the making and handling of mixed sulphuric and nitric acids for nitration purposes, and will prove of especial interest to those connected with the manufacture of explosives.

Chapter I. deals with the production of nitrate of soda from the natural deposits in Chili, and with the various main processes of extraction and separation. Chapter II. deals with the manufacture of nitric acid by distillation of Chili saltpetre with sulphuric acid, and includes detailed particulars furnished by several large works of general working methods; this chapter also discusses the concentration and recovery of weak nitric acid, and the costs of working, and a valuable section is contributed by Dr. G. A. Welsh, the Medical Superintendent at the Gretna Factory, on acid burns, poisoning, and general medical equipment. Chapter III. deals with methods of analysis, and nitre-cake; Chapter IV. with denitration of waste acids; Chapter V. with the mixing of acids for nitration purposes and the methods of doping and production of mixed acids of requisite strengths; Chapter VI. deals with chemical and physical properties of nitric acid; Chapter VII. with those of industrial nitrates and production of same; while Chapter VIII. reverts to mixed acids and the calculation of mixes for nitration purposes.

The book is well written, and the grouping of references, patents and analyses together at the end of each section will be found useful when any particular point is being looked up. The emphasising and frequent repetition of somewhat obvious details will be valuable to a novice, but are rather too prominent in a work of this class, which, on the whole, should be a valuable reference book for the technical man. The sections dealing with the mixing and doping of acid mixes, and the mathematical factors involved are very fully written, are set out clearly, and should prove very useful to those who have to deal with acid mixing; also the acid balance at the end of the book is a valuable example of how the rather complicated requirements of an explosives factory in regard to acids can be worked out and covered.

It would have been an advantage if all the methods of analysis had been grouped together in one chapter, as this would have avoided repetition and cross references. Also, formulæ should be given, to make tests which are not obvious, perfectly clear, as on p. 31. (Test for Iodates), and on p. 244, (Test for Nitrous Acid).

There are very few misprints, but on p. 155 the author gives 80 per cent. nitric acid as being commonly produced from Glover tower acid and ordinary nitrate; this should be 80° Tw. or 65 per cent. In Chapter VII. the author deals with the manufacture of ammonium nitrate, and refers to the U.S.A. plant at Perryville; it would have been useful if more details could have been given of the very large plants erected for this purpose in this country, especially of the Swindon factory, as, doubtless, the industrial application of the phase rule to mixed solutions will be of increasing importance in years to come.

The collection of patents and extracts therefrom in reference to the subjects treated take up a considerable portion of the book, and in future editions might, with advantage, be further curtailed; in fact, the whole book could be further condensed without seriously detracting from its utility.

The author has compiled a very full and extensive text-book which is a valuable record of experience gained on large scale plant for the manufacture of nitric acid. It will be valuable to both the student and the technical man, and should be included in every technical chemical library. H. J. BAILEY.

Publications Received.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. Vol. I. 5th Edition. J. & A. Churchill. **1924**. Price **30**s. net.

CANNED FOODS IN RELATION TO HEALTH. By W. G. SAVAGE, M.D. Cambridge University Press. Price 8s. **6d**. net.

THE CHEMISTRY AND PHYSICS OF CLAYS. By A. B. SEARLE. Ernest Benn Ltd. Price **55**s. net.

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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An extraordinary meeting was held at the Institute of Chemistry on January 16, 1924. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Hugh Browning Brown, A.I.C., Sidney Augustus de Lacy, A.I.C., A.M.I.Chem.E., Joseph Henry Lane, F.I.C., B.Sc. (Lond.), Leslie Herbert Lampitt, D.Sc. (Birm.), F.I.C., Reginald Francis Moon, B.Sc. (Bristol), Maximilian Nierenstein, D.Sc. (Geneva), Ph.D. (Berne), William Simpson Shaw, M.Sc., A.I.C., and Robert Norman Wright, A.R.C.S., B.Sc. (Lond.), A.I.C.; Misses Phyllis Honor Price, B.Sc. (Bristol), and Mabel Suzanne Lavinia Snelus, A.I.C.

Certificates were read for the second time in favour of Messrs. Thomas Francis Doyle, George Hogan, F.I.C., Frank Knowles, Archibald Knox, A.I.C., Charles Roger Middleton, B.Sc. (Lond.), A.R.C.S., A.I.C., and Harold Richard Read, A.I.C., A.R.C.Sc. (Dublin).

The following were elected members of the Society:—Messrs. Hubert Thomas Stanley Britton, B.Sc. (Bristol), M.Sc. (Lond.), F.I.C., and Robert Charles Frederick.

The Estimation of Nitrogen in Coal.

By W. DONOVAN, M.Sc.

(*Read at the Meeting, November* 7, 1923.)

In 1915, A. C. Fieldner and C. A. Taylor made a comparison of various modifications of the Kjeldahl method with the Dumas method, as applied to the estimation of nitrogen in coal. By the use of the Gunning modification of the Kjeldahl method, and with prolonged heating in the Dumas method to ensure complete combustion of the coal, the difference in average values by both methods was 0·10 per cent. The Dumas results were the higher.

E. Terres, H. Fleischer, and others, in 1919, also investigated the estimation of nitrogen in coal and coke. They found that both methods, as usually conducted, gave low results, the Kjeldahl method by loss of gaseous nitrogen, the Dumas owing to incomplete combustion. A modification of the Kjeldahl method was devised so that the gases evolved in the oxidation of the coal could be collected and the nitrogen, which was invariably present, recovered. The Dumas method was modified to permit of the generation inside the combustion tube of a supply of oxygen in the final stages of the operation.

The results obtained were:

	Percentage of Nitrogen.				
	Peat.	Lignite.	Saar Coal.	Ruhr Coal.	Anthracite.
Kjeldahl	1·70	0·80	1·31	1·33	1·35
,, + free *N*	1·77	1·20	1·76	1·58	1·84
Modified Dumas	1·72	1·08	1·73	1·55	1·72

The Research Sub-Committee of the Gas Investigation Committee of the Institute of Gas Engineers, in a report on the Comparative Economics of Production, from Thermal and Chemical Standpoints, of different Grades of Gas, confirmed the work of Terres as regards the modified Dumas method, but reported adversely on the Kjeldahl method. Some published results were:

	Percentage of Nitrogen.		
	Coal.	Coke.	Tar.
Kjeldahl	1·41–1·67	1·23–1·56	0·83–1·09
Modified Dumas	1·91–2·06	1·74–1·87	1·58–1·79

The procedure for the Dumas method as described in the report makes provision for the removal of carbon monoxide and dioxide, but apparently not for methane. The results are not very concordant and are probably somewhat high.

In the course of a series of analyses of New Zealand coals it was necessary to estimate nitrogen, and with the object of avoiding the more tedious combustion method it was decided to ascertain the relation, if any, between results by the two methods, and whether by applying a correction to the Kjeldahl figure the true result could be obtained. The combustion method followed was that recommended by the Association of Official Agricultural Chemists, as published in *Methods of Analyses*, 2nd edition, page 9. The operation is carried out in a previously evacuated tube, and the gaseous products of combustion removed by means of a mercury pump. This does away with the use of carbon dioxide gas for sweeping out the tube. In addition, the end of the tube was filled with potassium chlorate, to provide oxygen for the completion of the combustion, as recommended by Terres.

A combustion tube, 15–17 mm. internal diameter and 80 cm. long, was sealed at one end and filled as follows:

(A), Ten grms. of potassium chlorate, a small plug of ignited asbestos, then (B), 10 cm. of broken pumice, 4 to 5 mm. in diameter, to insulate the potassium chlorate from the heat in the first part of the combustion. Then (C), 4 cm. of powdered copper oxide, and (D), the combustion mixture of 1 grm. of coal and 25 grms. of finely powdered copper oxide, prepared from copper wire oxide. Then (E), 1 cm. of fine copper gauze, and (F), 15 cm. of copper wire oxide. (G), 5 cm. copper gauze, and (H), another 10 cm. of copper wire oxide.

The combustion tube was connected with a distilling flask of 100 c.c. capacity, containing 15 grms. of soda lime, moistened slightly and spread over glass wool, to increase the effective absorption surface. This in turn led to a Sprengel pump. The tube and absorption vessel were evacuated and the wire copper oxide and gauze (F, G and H) heated until they were red hot. The heating was gradually extended, until the mixture of coal and copper oxide, D, was reached. This was heated carefully and the products of combustion, passing over the hot copper oxide, were converted mainly into carbon dioxide, and were absorbed by the soda lime. When the evolution of gas had apparently ceased, the tube was evacuated, and the gas collected in a suitable receiver.

The potassium chlorate A was then heated carefully, to generate a slow, steady stream of oxygen. The copper reduced in the first stage of the combustion was re-oxidised and any unconsumed carbon completely burned, the reaction causing a glow by which its progress through the mixture could be followed. When the copper gauze, E, showed signs of oxidation heating was discontinued, and the tube again evacuated. The gas collected was mixed with that obtained in the first stage of the combustion, and the whole transferred to the absorption vessel of a Bone and Wheeler gas analysis apparatus, which was calibrated to measure volume. Oxygen and carbon dioxide were absorbed by means of alkaline pyrogallol, and the volume of unabsorbed gas measured in the water-jacketed tube, over mercury. The volume varied from 20 to 35 c.c. An aliquot portion was exploded with excess of oxygen and electrolytic gas in the usual manner, to remove carbon monoxide and hydrocarbons, the volume of the residual nitrogen was measured, and from it the total volume of nitrogen was obtained by calculation.

That considerable amounts of hydrocarbons may escape oxidation in the combustion tube is shown by the following analyses:

A. Gas resulting from combustion of one grm. of sugar.

B. Gas resulting from combustion of one grm. of coal.

(Gases were freed from oxygen and carbon dioxide prior to analysis.)

	A.	B.
Carbon monoxide	5·2	7·8
Methane	61·9	19·7
Ethane	12·0	4·7
Nitrogen	20·9	67·8
	100·00	100·00

(The nitrogen in A was derived from the copper oxide used.)

It is apparent that, unless provision is made for the removal of hydrocarbon gases, the results for nitrogen may be considerably in excess of the truth.

It is necessary to apply a correction for nitrogen in the copper oxide mixed with the coal. Fieldner and Taylor (*infra*) found that all copper oxide procurable by them contained nitrogen that was not removed by heating *in vacuo* for several hours, but was liberated under the conditions of the experiment. The wire form was much purer than either the lump or powder form.

Blank combustions were therefore carried out with three different samples of copper oxide, with the use of pure sugar instead of coal. The results obtained were:

	Nitrogen c.c.
30 grms. copper oxide powder (Merck)	4·5
30 grms. copper oxide lump form (Merck)	2·42
30 grms. wire oxide powdered (British Drug Houses)	0·50

These confirm Fieldner and Taylor's observations.

Powdered wire oxide ground to pass a 60-mesh sieve was therefore used for mixing with the coal, a correction being applied for the nitrogen content.

A trial estimation in which a mixture containing 0·15 grm. of diphenylamine and 0·85 grm. of sugar was used gave nitrogen 1·256 per cent. (theoretical value, 1·243 per cent.).

For the Kjeldahl process 1 grm. of the coal was taken, 15 grms. of potassium sulphate, and 30 c.c. of sulphuric acid, with 0·4 grm. of mercury as catalyst. The mixture was heated somewhat rapidly at first, and then the heat regulated so that clearing took place in about an hour. The heating was continued for a further three hours. After precipitation of the mercury with sodium sulphide, the ammonia was distilled off into 25 c.c. of 0·1 *N* sulphuric acid. When the mercury was omitted, or when smaller amounts of potassium sulphate were used, less satisfactory results were obtained.

Nitrogen was estimated by both methods in five coals which were known to differ considerably in general properties and in nitrogen content. These had the following proximate analyses:

	1. Per Cent.	2. Per Cent.	3. Per Cent.	4. Per Cent.	5. Per Cent.
Fixed carbon	61·80	56·78	57·37	42·44	43·68
Volatile hydro-carbons	32·75	38·38	39·92	39·06	40·55
Water lost at 100° C.	0·75	2·47	1·08	16·00	11·41
Ash	4·70	2·17	1·63	2·50	4·36
	100·00	100·00	100·00	100·00	100·00
Sulphur, per cent.	0·76	0·27	5·33	0·26	0·96

No. 1 was a bituminous coal, low in sulphur, and yielding a highly swollen coke. No. 2 was similar, but the coke was only moderately swollen. No. 3 was very high in sulphur. Nos. 4 and 5 were brown coals.

The results for nitrogen were:

	PERCENTAGE OF NITROGEN.				
No. of Coal.	Dumas.		Kjeldahl.		Difference of Average.
1.	2·05 2·07 2·09	Average 2·07	1·92 1·89 1·85 1·88	Average 1·89	0·18
2.	1·77 1·74	Average 1·755	1·58 1·62 1·54 1·60	Average 1·585	0·17
3.	1·02		0·88 0·83 0·84	Average 0·85	0·17
4.	1·08		0·88 0·88 0·85 0·86	Average 0·87	0·21
5.	1·32 1·29	Average 1·305	1·09 1·11 1·05 1·05	Average 1·075	0·23

The agreement of duplicates by the Dumas method was very good, and by the Kjeldahl method, satisfactory, though not quite so good.

The loss of nitrogen by the Kjeldahl process specified is fairly constant, being 0·17 per cent. for the bituminous coals, and 0·22 per cent. for the brown coals. It could be taken as 0·20 per cent. for both types of coal without serious error.

SUMMARY.—Nitrogen was estimated in five coals by the Dumas method. Complete combustion was assured by a supply of oxygen in the final stage. The greater part of the carbon dioxide formed was absorbed within the apparatus. By means of explosion and absorption in a Bone and Wheeler gas analysis apparatus all hydrocarbons and other extraneous gases were eliminated. The final volume of nitrogen was measured over mercury, in a water-jacketed tube in the same apparatus. The results were corrected for nitrogen in the copper oxide used.

Nitrogen was estimated in the same coals by the Kjeldahl-Gunning process, with the use of mercury as a catalyst.

The Kjeldahl results, plus 0·2 per cent., would give the correct percentage of nitrogen in the coals examined.

REFERENCES.

A Comparison of Various Modifications of the Kjeldahl Method with the Dumas Method of determining Nitrogen in Coal. A. C. FIELDNER and C. A. TAYLOR, *J. Ind. Eng. Chem.*, 1915, 7, 106.

Estimation of Nitrogen in Coal and Coke. E. TERRES, H. FLEISCHER, and others. *J. Gasbeleucht*, 1917, 52, 173–177; 192–200.

(Abstract in *J. Soc. Chem Ind.*, 1919, 30, 399A.)

Fourth Report of the Research Sub-Committee of the Gas Investigation Committee of the Institute of Gas Engineers. Appendix II. Methods of Analysis. *Gas Journal*, 1920; 150, 624.

DOMINION LABORATORY,
WELLINGTON, NEW ZEALAND.

DISCUSSION.

Mr. G. RUDD THOMPSON said that he considered the paper a most valuable contribution and most explicit. The importance of this question from a technical point of view to-day was the yield of ammonium sulphate the gas-makers were going to get from their coal, and, in his opinion, the paper solved a somewhat difficult question. With the Kjeldahl method one got somewhere about 0·2 per cent. less nitrogen than with the Dumas method, but that nitrogen was not recoverable in the by-product plant. The author had proved this low result to be due to loss of gaseous nitrogen. Further, to obtain his results the author had taken a wide range of coals which would very reasonably cover the average coals used in by-product plants in this country.

Mr. G. N. HUNTLY noticed that the author had made no mention of the formation of nitric oxide. The addition of electrolytic hydrogen might partially remove it, but he felt the paper would have been more satisfactory had it been mentioned. The presence of methane seemed to have been noticed, and if one could arrange to pass the gas backwards and forwards, as in a gas-analysis apparatus, one would get rid of it, but not otherwise. Another point was that when working in a vacuum one had to look out for leaks. An error of 0·2 per cent., due to occluded nitrogen, did not sound much, but it really was serious.

Mr. R. L. COLLETT asked whether the author had attempted to find out—by collecting the gas evolved by his Kjeldahl apparatus—if the 0·2 per cent. loss was due to loss of gaseous nitrogen. He suggested that possibly the digestion stage of the process was not carried to completion.

Mr. RAYMOND ROSS enquired what gases were occluded by coal, and how much actual nitrogen was occluded. It was well-known that very different results were obtained, as to the amount of ammonium sulphate, according to the method of carbonisation employed. He asked whether lead chromate could not be used for all ordinary combustions of that kind instead of copper oxide; personally, for ordinary combustion, he preferred rolls of oxidised copper gauze to granular copper oxide.

Dr. H. E. COX said that he had, for some years, used copper gauze which had been oxidised in the manner now put forward by the author. In his experience the amount of nitrogen in coal was generally about 1·4 per cent.; he considered the author's result of 1·7 per cent. too high; the Dumas method often gave too high results on account of unburnt gases, and the gas evolved in the Kjeldahl digestion was not nitrogen derived from the coal substance.

Mr. RUDD THOMPSON, replying, said that many years ago methane and ethane were occasionally found sporadically, and it was rather strange that no one had taken the trouble to find out the reason of their presence. Although the joint between the combustion tube and the Sprengel shewn in Mr. Donovan's sketch was a cork joint, very probably in actual use the combustion tube was drawn out, so as to avoid leakage.

Effects of Storage on Artificially Polluted Waters.

BY ROBERT C. FREDERICK.

(*Read at the Meeting, March,* 1923.)

IN the course of an extensive experience in the analysis of water it became increasingly evident that many of the orthodox ideas regarding the interpretation of water analysis results were erroneous. Doubts as to the correctness of some of these beliefs have already been expressed elsewhere (1), and this paper contains an account of a subsequent detailed investigation into the chemical changes which occur in excretally polluted water, and the significance of these in the interpretation of the results of an analysis.

In addition to enabling a correct interpretation of the analytical results for a sample to be made when the analysis had been conducted soon after collection, the research had for a further object the determination of the utility of an analysis when a long period had elapsed between collection and examination. This study has been made on samples only and, though the conditions with these are not exactly comparable with those in the case of a water supply, doubtless the results also give some indication of the probable occurrences *in situ.* The findings have been co-ordinated with statistics of the results obtained by the author in the analysis of nearly 1000 samples from every kind of supply throughout the British Isles; the samples were limited to this number so as to include only those which had been examined in recent years by a technique improved by the latest modifications.

The investigation was made with reference only to the substances whose presence is indicative of excretal pollution, *i.e.* free ammonia, albuminoid ammonia, nitrites, and nitrates. The chlorine content, the value of which in this respect has already been discredited, has received some minor consideration. The writer has, elsewhere, detailed his reasons for abandoning the "oxygen absorbed from potassium permanganate" process in the routine examination of water (2). The analytical processes employed were Wanklyn's for free and albuminoid ammonia (3), modified Lombard's for nitrites (4), and Frederick's for nitrates (5).

The general principle of the research was the preparation of bulk quantities of polluted water, which were then divided into samples of exactly similar original composition and stoppered bottles were filled with them. These were set aside under pre-determined conditions and their contents analysed after various periods of storage up to 100 days.

The water which formed the basis of the majority of these samples was ammonia-free water distilled from an all-glass apparatus. To reproduce the character of natural water a quantity of fresh suspension of soil taken from underneath growing turf was added, after rough filtration through muslin and, in some series, there were added salts commonly present in natural potable waters. Details of each series of samples are given under the headings below. In every instance free ammonia and albuminoid ammonia is expressed as NH_3, and nitrites and nitrates as N.

SAMPLES STORED IN THE DARK AT ROOM TEMPERATURE (SERIES L, M, AND O.—The object of these series was to determine comparatively the changes which took place when the same water was polluted with urine, (L); with an equal quantity of fæces, (M); and with the same weight of a mixture of equal parts of urine and fæces, (O).

Series L.—A large aspirator bottle, graduated at 2500, 5000 and 7500 c.c., was filled to about 6000 c.c. with the distilled water, which was aerated by shaking; 100 c.c. of mineral salts partly dissolved and partly in suspension were then added, with shaking, and then 5 c.c. of soil suspension. The whole was mixed. The 100 c.c. of mineral salts mixture contained: Sodium chloride, 0·225 grm.; calcium chloride, 0·750 grm.; magnesium chloride, 0·150 grm.; calcium carbonate, 0·075 grm.; calcium sulphate, 0·750 grm.; and calcium oxide, 0·225 grm. The samples, as finally prepared, had a total hardness of 10·7 parts per 100,000. Ten c.c. of a dilution of urine, of original specific gravity of 1·030 (5 c.c. with distilled water to 100 c.c.) from a healthy human, equal to 0·5 c.c. of urine (0·515 grm.), were then added, and also (to approximate more closely the conditions of a natural water) 1250 c.c. of an upland surface water of good quality, collected three weeks previously, containing no free ammonia, 0·006 part per 100,000 of albuminoid ammonia, no nitrites, and only a minute trace of nitrates. The whole was made up to 7500 c.c. and thoroughly shaken; 2500 c.c. of this mixed water were removed for the preparation of Series O, and the remaining 5000 c.c. were made up to 7500 c.c. with the distilled water and mixed. This water was that used in Series L, and was polluted with urine to the extent of approximately 1 part in 22,000 by weight. It was put aside in the dark in eleven 500 c.c. and six 250 c.c. bottles, and a daily record was made of the average temperature prevailing in the storage cupboard during the previous 24 hours.

Series M.—This series was prepared and stored exactly as in series L, except that the pollution was by an equal weight of fæces, instead of urine, 0·515 grm. being added after disintegrating 5·150 grms. by shaking with beads and water, and taking 25 c.c. after dilution to 250 c.c. It is to be noted that this degree of pollution with fæces imparted a distinct turbidity and colour to the water.

Series O.—This series was prepared and stored as in series L and M, but contained a mixture of half the quantity of urine in the former together with half the quantity of fæces present in the latter series; the total degree of pollution was therefore the same as in the two previous series.

The analytical results obtained in these series are shown in Table I.

In the first place it is to be noted that the chemical changes in samples take place comparatively slowly, and that quite different results are obtained according to whether the pollution is urinary, fæcal, or a mixture of both. It has always been considered that the chemical evidence of excretal pollution in water samples disappeared so rapidly that if much delay occurred between collection and analysis the results obtained were of little or no value, and that this was also the case, even during a short delay, if the samples were not kept cold. The analytical results

TABLE I. Series L, M and O.

Analytical results after various periods of storage of samples polluted with urine (L): fæces (M): urine and fæces (O). Parts per 100,000.

Number of Days Stored	Average Temperature of Storage space to date	L				M				O			
		Free Ammonia	Albuminoid Ammonia	Nitrites	Nitrates	Free Ammonia	Albuminoid Ammonia	Nitrites	Nitrates	Free Ammonia	Albuminoid Ammonia	Nitrites	Nitrates
Immediately	—	0·0064	0·0164	0·0001	0·0040	0·0008	0·0388	0·0002	0·0040	0·0026	0·0220	0·0002	0·0040
4	18·1° C.	0·0068	0·0158	0·0001	0·0040	0·0011	0·0404	0·0002	0·0040	0·0044	0·0236	0·0002	0·0040
11	16·9 ,,	0·0108	0·0140	0·0001	0·0040	0·0148	0·0246	0·0002	0·0040	0·0096	0·0202	0·0002	0·0040
18	16·8 ,,	0·0108	0·0138	0·0001	0·0040	0·0232	0·0250	0·0002	0·0040	0·0208	0·0182	0·0002	0·0040
21	16·9 ,,	—	—	0·0001	—	—	—	0·0003	—	—	—	0·0003	—
25	16·0 ,,	0·0144	0·0156	0·0001	0·0040	0·0238	0·0256	0·0003	0·0040	0·0216	0·0168	0·0003	0·0040
31	15·3 ,,	—	—	0·0001	—	—	—	0·0005	—	—	—	0·0003	—
35	15·2 ,,	0·0144	0·0144	0·0001	0·0040	0·0272	0·0244	0·0007	0·0040	0·0224	0·0206	0·0005	0·0040
39	15·1 ,,	—	—	0·0001	—	—	—	0·0072	—	—	—	0·0015	—
45	15·0 ,,	0·0168	0·0138	0·0001	0·0040	0·0280	0·0224	0·0010	0·0040	0·0188	0·0212	0·0076	0·0040
49	15·0 ,,	—	—	0·0010	—	—	—	0·0240	—	—	—	0·0070	—
54	15·2 ,,	—	—	0·0100	—	—	—	0·0160	—	—	—	0·0120	—
57	15·3 ,,	0·0160	0·0162	0·0004	0·0040	0·0216	0·0236	0·0047	0·0060	0·0152	0·0198	0·0152	0·0080
63	15·5 ,,	0·0140	0·0154	0·0002	0·0040	0·0004	0·0212	0·0160	0·0080	0·0000	0·0200	0·0001	0·0080
67	15·7 ,,	0·0085	—	0·0043	0·0040	—	—	0·0000	0·0080	—	—	0·0000	0·0120
79	15·8 ,,	0·0188	0·0174	0·0012	0·0060	0·0000	0·0142	0·0000	0·0100	0·0000	0·0154	0·0000	0·0140
100	15·8 ,,	0·0168	0·0152	0·0010	0·0060	0·0000	0·0140	0·0000	0·0120	0·0000	0·0128	0·0000	0·0400

of these series completely disprove this view and, moreover, show that, if pollution of the supply has only been very recent, the evidence in samples would be more pronounced if the analysis were actually delayed for a considerable period.

The statement is reiterated in the literature that animal pollution of water is indicated by a high free and a low albuminoid ammonia content, *i.e.* the quantity of albuminoid ammonia is only a small fraction of that of the free ammonia. The statement appears to be founded on isolated analyses of samples of sewage and sewage effluents. The fallacy is apparent when it is seen that the amounts and proportion of the free and albuminoid ammonia are constantly varying from the moment pollution takes place. In point of fact, the quantity of albuminoid ammonia is at no time a small fraction of that of the free ammonia; it is seldom appreciably less, is frequently in excess, and at some periods very markedly in excess, as will be seen from the table below showing the proportion of albuminoid ammonia to one of free ammonia at different periods.

TABLE II. SERIES L, M, AND O.

Proportion of albuminoid ammonia to one part of free ammonia after various periods of storage.

		Days stored									
Series	Immediate	4	11	18	25	35	45	57	63	79	100
L.	2·6	2·3	1·3	1·3	1·1	1·0	0·8	1·0	1·1	0·9	0·9
M.	48·5	36·7	1·7	1·1	1·1	0·9	0·8	1·1	53·0	—	—
O.	8·5	5·4	2·1	0·9	0·8	0·9	1·1	1·3	—	—	—

Pollution of water with either urine or fæces causes an addition of a marked amount of albuminoid ammonia, which is in evidence from the outset before the production of free ammonia has fully developed, and a large proportion remains even when the free ammonia and nitrites have been converted entirely into nitrates. The albuminoid ammonia is therefore of primary importance as an indicator of excretal pollution.

While it is probable that the production of nitrites is accelerated *in situ* under the conditions of certain water supplies, the results obtained in these series show that, in samples, nitrites are only produced a considerable time after the addition of excretal matter, and it follows, then, that the interpretation of their presence as indicating very recent pollution is unwarranted. Taking 0·0005 parts per 100,000 as distinct evidence by the Lombard test, it will be seen that nitrites did not appear until the 49th day in Series L, the 31st day in Series M, and the 35th day in Series O. In elaboration of this view, it is to be noted that nitrites do not indicate very recent pollution, as their production in quantity is nearly always subsequent to a decrease of the free ammonia content, and this only commences after a considerable period. Similarly, the production of nitrates follows a decrease in the nitrite content, and pollution indicated by nitrates is certainly of remote date. The progressive nature of these changes is demonstrated by the remote date.

It is generally understood that the presence of nitrites is of very short duration, almost fugitive, but the figures in Table I. show this belief also to be untenable.

Adopting the periods and minimum figure above, they were present for 51, 32 and 22 days in Series L, M and O respectively. Shorter periods have been noted in some other series. It is probable that the misconception has originated by the testing of portions from the one bottle instead of single portions from separate full bottles. The former method is fallacious as it does not imitate the conditions of water examination practice, and that it has a destructive action on the nitrite content is seen by the results of Series E.

SERIES E.—In this series the sample water was prepared as in those discussed previously, with 6 litres ammonia-free distilled water and 2 litres tap water of excellent quality from the chalk and free from nitrites, 0·5 c.c. soil suspension, 0·5 c.c. urine, and 2·0 c.c. emulsion of fæces. The whole was thoroughly mixed and divided into stoppered bottles as follows:

E.1. Seven 500 c.c. filled full.
E.2. One winchester quart filled to shoulder.
E.3. One winchester quart filled full.

A separate bottle from E.1. was tested for nitrites at weekly intervals dating from the day of preparation, the Winchester quart E.2 was tested every few days (sometimes daily), and the Winchester quart E.3 was similarly tested after remaining sealed for 16 days. The quantity of nitrite present at various periods is shown in Table III.

TABLE III. SERIES E.

Analytical results; amount of nitrites present in separate sealed samples (E.1), compared with that present in samples of the same water, repeatedly tested (E.2 and E.3), parts per 100,000.

Days stored	Average temperature to date of analysis	Nitrites E.1	E.2	E.3
Immediate.	—	0·0000	0·0000	
1	20·7° C.	—	0·0000	—
2	20·1 „	—	0·0000	—
3	19·2 „	—	0·0000	—
4	19·0 „	—	0·0001	—
5	18·9 „	—	0·0001	—
6	18·9 „	—	0·0001	—
7	18·9 „	0·0001	0·0001	—
8	18·8 „	—	0·0001	—
9	18·8 „	—	0·0001	—
10	18·5 „	—	0·0001	—
11	18·3 „	—	0·0001	—
13	18·1 „	—	0·0001	—
14	17·9 „	0·0002	0·0001	—
16	17·8 „	—	0·0001	
17	17·9 „	—	0·0001	0·0002
18	17·8 „	—	0·0001	0·0002
21	17·8 „	0·0010	0·0001	0·0002
24	17·9 „	—	0·0001	0·0002
27	18·0 „	—	0·0001	0·0004
28	18·0 „	0·0010	0·0001	0·0007
30	17·9 „	—	—	0·0010
32	17·9 „	—	0·0007	0·0030
34	17·8 „	—	0·0020	0·0060
35	17·8 „	0·0300	—	0·0100
37	17·8 „	—	—	0·0100
42	18·0 „	0·0300	0·0060	0·0100

In resuming the general discussion it has to be pointed out that the alleged short duration of the presence of nitrites is responsible for the teaching that quantitative examination for them is not worth while; this premise is at fault, for the real explanation is the marked fluctuation in quantity.

It is noteworthy that in the case of very recent pollution, particularly urinary, the free ammonia obtained by distillation is not infrequently evolved in a large number of fractions, each of which, after the first few, contains the same amount as, or very slightly less than, that immediately preceding; in samples in which pollution is more remote the free ammonia is completely evolved in a small number of fractions which progressively show a distinct fall in amount. This phenomenon is illustrated in Table IV., containing the results obtained with Series F—a water polluted with approximately one part of urine in 15,000; the quantity of the sample distilled was 250 c.c. in each case and the speed of distillation was approximately the same throughout.

TABLE IV. SERIES F.

Free ammonia contained in 50 c.c. fractions on distilling 250 c.c. of sample; amount expressed in terms of c.c. of standard ammonium chloride solution (1 c.c. = 0·01 mgrm NH_3).

Days stored.	Fraction. 1st	2nd	3rd	4th	5th	6th	7th	8th	9th	10th	Total
1	1·0	0·7	0·35	0·3	0·2	0·15	0·15	0·1	0·1	0·05	3·10
5	1·0	0·9	0·8	0·4	0·3	0·2	0·1	0·05	—	—	3·75
33	2·5	2·1	1·2	0·7	0·5	0·1	—	—	—	—	7·10
39	2·5	2·1	1·6	0·7	0·4	0·1	—	—	—	—	7·40

It will be seen that 1 day and 5 days after pollution, the free ammonia was evolved in 10 and 8 fractions respectively, whilst after 33 and 39 days it was completely evolved in 6 fractions, though double the amount of ammonia was present.

The phenomenon also aids in distinguishing very high free ammonia due to recent gross pollution from the high amounts met with in some waters due to the reduction of nitrates. An example is given in Table V. D was a water polluted with urine to the extent of one part in 2500, and No. 720 was from a well in Lincolnshire, 100 feet deep, free from excretal pollution. Both samples were analysed after 2 days' storage.

TABLE V.

Free ammonia contained in 50 c.c. fractions on distilling 100 c.c. of sample; amount expressed in terms of c.c. of standard ammonium chloride solution (1 c.c. = 0·01 mgrm. NH_3). D was a water containing recent urinary pollution; No. 720 a water free from excretal pollution.

	Fractions. 1st	2nd	3rd	4th	5th	6th	7th	8th	9th	10th	11th	12th	13th	14th	15th	16th	17th	18th	19th	20th	Total
D.	3·0	3·0	2·0	1·5	1·0	0·8	0·7	0·6	0·5	0·4	0·4	0·5	0·5	0·5	0·4	0·4	0·5	0·5	0·4	0·4	18·0
720	10·0	5·5	2·5	1·1	0·6	0·2	0·1	—	—	—	—	—	—	—	—	—	—	—	—	—	20·0

Recent pollution is indicated by distinct evidence of free ammonia, particularly if evolved in the manner described above (sample D), associated with about an equal amount of albuminoid ammonia; if exclusively fæcal, very recent pollution may not be evident by this figure, although the relatively great albuminoid ammonia content is a compensating indication.

In considering the occurrence of free ammonia, albuminoid ammonia, nitrites, and nitrates in water as indicators of excretal pollution, it has to be remembered that all may be derived from harmless sources. In rain or distilled waters, or those in which reduction of nitrates by contact with metals or particular strata can be presumed, free ammonia and nitrites have no significance, and in water from newly constructed wells or containing decaying vegetable matter, they have a significance only when present in marked quantity. In upland surface waters the albuminoid ammonia has no significance, and in waters which are supplied from small storage tanks or cisterns this figure has commonly no import as it is derived from accumulating débris. In waters from certain strata, *e.g.* the chalk, nitrates have no significance.

In parenthesis some remarks may be made here regarding decaying vegetable matter as a source of free ammonia and nitrites. The high figures which can be obtained from this source alone, and cause an erroneous attribution to the presence of excreta, do not appear to have received adequate recognition. In this connection the results of Series W.1 and W.2 are of interest. W.1.—Three 500 c.c. stoppered bottles were filled with tap water of good quality from the chalk. Freshly gathered green grass was thoroughly washed ammonia-free and pieces of blade (6·0 by 0·3 cm.) were cut. Into the first bottle one blade was placed, and in the second, two; the third bottle contained no grass and acted as a control. These were put aside in the dark until the grass had decayed, as evidenced by the almost complete disappearance of green colour (10 days), and were then analysed for free ammonia, albuminoid ammonia, and nitrites. *W*.2.—This Series was prepared exactly as W.1, except that ammonia-free distilled water, to which a trace of fresh soil suspension had been added, was employed. In this case decay was not sufficiently far advanced until after 32 days. The same determinations were then made as above. The results are shown in Table VI. The marked amount of free ammonia and nitrites which may be derived from decaying vegetable matter is evident.

TABLE VI.

Analytical results of samples showing the effect of the presence of decaying vegetable matter.

Water.	Free Ammonia. Parts per 100,000.			Albuminoid Ammonia. Parts per 100,000.			Nitrites. Qualitative test.		
	1 Blade	2 Blades	Control	1 Blade	2 Blades	Control	1 Blade	2 Blades	Control
Tap (W.1)	0·0234	0·0450	0·0002	0·0050	0·0084	0·0020	Present	Present	Absent
Distilled(W.2)	0·0250	0·0506	0·0005	0·0042	0·0032	0·0008	Present	Present	Minute trace

A small quantity of free ammonia may also be derived from undecayed vegetable matter if contained in the portion distilled for this determination. An extreme case (Series *W*.3) is cited. To each of two quantities of 500 c.c. of ammonia-free distilled water 25 half blades of freshly washed grass were added. In the one sample, decanted from the grass, free ammonia was absent; in the other, distilled with the grass, a result of 0·0150 parts per 100,000 was obtained.

To resume, in the circumstances detailed in the penultimate paragraph above it is impossible to fix limits, but, if the appropriate source can be excluded as the origin of the presence of any of the substances enumerated, it is possible to formulate limits which, if exceeded, afford evidence of excretal pollution. I consider the present accepted limits for free and albuminoid ammonia too wide, and adherence to these must inevitably result in passing as fit for potable purposes a considerable proportion of water samples which are undoubtedly contaminated with excreta. In this connection the figures obtained (Tables VII. and VIII.) in the statistical investigation referred to in the second paragraph of the introduction are illuminating; in all cases the chemical opinion as to the purity of the samples was formed with full knowledge of the supply and was usually confirmed by independent bacteriological examination.

TABLE VII.

Amounts of free ammonia and nitrites in consecutive samples of water considered, in view of analytical results and topographical conditions of supply site, (1) free from excretal pollution; (2) excretally polluted; excluding those which could contain either free ammonia or nitrites from other than excretal sources.

Constituent	Parts per 100,000	Percentage of samples containing free ammonia and nitrites in excess of amount stated. 1. Free from pollution	2. Polluted
Free Ammonia	Exceeding 0·001	15	84
	„ 0·002	3	73
	„ 0·003	Nil	68
	(Average amount = In unpolluted samples 0·0005 In polluted samples 0·0127)		
Nitrites	Absent	95	3
	Exceeding 0·0001	Nil	86

TABLE VIII.

Amount of albuminoid ammonia in consecutive samples of water considered, in view of analytical results and topographical conditions of supply site, (1) free from excretal pollution, (2) excretally polluted; excluding those which could contain albuminoid ammonia from other than excretal sources.

Albuminoid Ammonia Parts per 100,000	Percentage of samples containing albuminoid ammonia in excess of amount stated. (1) Free from pollution	(2) Polluted
Exceeding 0·002	50	100
„ 0·004	25	98
„ 0·006	9	98
„ 0·008	Nil	84
(Average amount = In unpolluted samples 0·0027 In polluted samples 0·0154)		

It will be seen that, of waters considered fit for potable purposes, none contained free ammonia in excess of 0·003, albuminoid ammonia in excess of 0·008, or nitrites in excess of 0·0001 parts per 100,000, after excluding samples which could have derived these substances from other than excretal sources. Nitrates should not exceed 0·050 parts per 100,000. These then are the limits which, if exceeded and a non-excretal origin can be excluded, I submit provide definite evidence of excretal pollution. Considered with the reservations detailed above, it is not necessary that all these figures should be exceeded to cause suspicion, a distinct excess of any one is sufficient, but the occurrence of only a single abnormality is uncommon. Attention is drawn to the large proportion of unpolluted waters in which the analytical results did not even approach these limits. It will be seen that in polluted waters the limit for free ammonia was not exceeded in 32 per cent. of cases, for albuminoid ammonia in 16 per cent., and for nitrites in 14 per cent. The large proportion of samples in which the free ammonia failed to give evidence of pollution is of interest; examples of such waters are given in Table IX.

The samples included are such as could naturally contain only a small amount of albuminoid ammonia, and the high figures obtained in this determination illustrate its value in detecting pollution. Such waters give free and albuminoid ammonia figures not dissimilar from those yielded by many pure upland surface waters, but the nitrite and nitrate results are totally different, and enable a clear distinction to be made; examples of such waters are shown in Table X.

Emphasis has been laid on the presence of albuminoid ammonia as an indicator of the entry of excretal pollution; its absence is equally valuable in showing freedom from pollution particularly in waters containing high free ammonia and nitrites from reduction of nitrates; examples of such waters are shown in Table XI.

In Series L, M and O, a determination was also made of the addition to the chlorine content caused by the degree of pollution employed (1 in 22,000). By estimating the chlorine in the urine and fæces used it was calculated that in the case of urine the increase was 0·15, and with fæces 0·007 parts per 100,000. It is evident, then, that the chlorine estimation is of no value for providing indication of fæcal and of little value for urinary pollution, unless this is in gross amount.

In a prior paper (6) the writer has given an account of the chemical changes which occur in samples similar to the foregoing under certain particular conditions; on exposure to light (Series C); when containing metallic impurities (Series H); when chlorinated (Series Q, R and S). It may be useful to make brief mention of the findings of this previous research here, as the effect of any of these conditions is very marked, and is an imperative consideration in the interpretation of an analysis.

Samples exposed to light (*Series C*).—It will be seen that exposure to light resulted in an enormous increase in the albuminoid ammonia content (due to growth of algae), but, for all practical purposes, there was no effect on the rate

TABLE IX

Analytical results of polluted waters containing a very small quantity of free ammonia, parts per 100,000.

	1	2	3	4	5	6	7	8	9	10
Free Ammonia ..	0·0003	0·0009	0·0002	0·0011	0·0007	0·0001	0·0017	0·0002	0·0000	0·0004
Albuminoid Ammonia	0·0195	0·0266	0·0196	0·0196	0·0186	0·0257	0·0148	0·0286	0·0174	0·0108
Nitrites ..	0·0010	0·0001	0·0002	0·0002	0·0001	0·0012	0·0011	0·0020	0·0014	0·0001
Nitrates ..	1·4280	2·1050	3·0770	0·3800	2·5000	0·5710	0·5710	0·4700	5·7140	2·0000

(1) Well, 22ft. deep (Kent); (2) Well, 40ft. deep (Lincolnshire); (3) Well, 18ft. deep (Norfolk); (4) Well, 12ft. deep (Cornwall); (5) Well, 20ft. deep (Cornwall); (6) Spring (Essex); (7) Spring (Cornwall); (8) Well, 40ft. deep (Norfolk); (9) Well, 32ft. deep (Devon); (10) Well, 22ft. deep (East Yorks.).

TABLE X.

Analytical results of unpolluted waters containing a large quantity of albuminoid ammonia, parts per 100,000.

	1	2	3	4	5	6	7	8	9	10
Free Ammonia ..	0·0008	0·0021	0·0004	0·0000	0·0018	0·0003	0·0012	0·0019	0·0015	0·0012
Albuminoid Ammonia	0·0130	0·0186	0 0127	0·0168	0·0172	0·0104	0·0136	0·0186	0·0256	0·0196
Nitrites ..	0·0000	0·0000	0·0000	0·0000	0·0000	0·0000	0·0000	0·0000	0·0000	0·0000
Nitrates ..	0·0040	0·0040	0·0060	0·0040	0·0040	0·0020	0·0140	0·0060	0·0160	0·0040

(1) Upland surface water (Linlithgowshire); (2) Moorland stream (Shetlands); (3) Burn (Orkneys); (4) Moorland stream (Orkneys); (5) Lake (Co. Donegal); (6) Mountain stream (Co. Cork); (7) Brook (Dorset); (8) Stream (Dumbartonshire); (9) Brook (Co. Cork); (10) Burn (Orkneys).

TABLE XI.

Analytical results of unpolluted waters containing a large quantity of free ammonia from reduction of nitrates, parts per 100,000.

	1	2	3	4	5	6	7	8	9	10
Free Ammonia ..	0·0974	0·1148	0·9840	0·0456	0·1154	0·0834	0·1054	0·0432	0·0576	0·0824
Albuminoid Ammonia	0·0040	0·0020	0·0033	0·0030	0·0012	0·0023	0·0035	0·0042	0·0016	0·0026
Nitrites ..	0·0001	0·0001	0·0001	0·0000	0·0001	0·0010	0·0000	0·0010	0·0010	0·0005
Nitrates ..	0·0020	0·0040	0·0050	0·0060	0·0020	0·0020	0·0040	0·0020	0·0040	0·0040

(1) Well, 400ft. deep (Essex); (2) Well, 50ft. deep (Hants.); (3) Well, 200ft. deep (Essex); (4) Well, 523ft. deep (Kent); (5) Bore well, 78ft. deep (Yorkshire); (6) Public supply (Essex); (7) Well, 300ft. deep (Hants.); (8) Spring (Orkneys); (9) Public supply (Kent); (10) Well, 400 ft. deep (Lincolnshire).

of production and the amount of free ammonia and nitrites. It has always been recognised that water samples must be stored in the dark, but the reason for this is that otherwise there is a fallacious increment to, not a decrement from, the indications of pollution as is commonly supposed.

Samples stored in the dark; effect of metallic impurities.—The presence of 0·1 part per 100,000 of copper (as sulphate) very greatly restrained the production of free ammonia and entirely inhibited its decomposition into nitrites and nitrates. The creation of evidence of original pollution has therefore been, to a great extent, prevented. In an investigation by Atkinson and the writer (7) on the influence of metallic impurities in water on its bacteriological content, it was found that the effect exerted by the four metals tested for in the routine examination of water was in the order—copper, zinc, lead, iron.

Samples stored in the dark; effect of chlorination.—Chlorination to the extent of 3·0 parts per million completely prevented, for all practical purposes, the production of free ammonia, nitrites, and nitrates, *i.e.* it prevented the formation of the evidence of pollution, and even one-quarter of this amount was only slightly less effective.

ROBERT C. FREDERICK.

ROYAL NAVAL MEDICAL SCHOOL,
ROYAL NAVAL COLLEGE, GREENWICH.

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7. ATKINSON, (E. L.) and ROBERT C. FREDERICK. *J. Roy. Naval Med. Service*, **1921**, **220**.

DISCUSSION.*

MR. E. HINKS: Some of the author's observations, and, still more, his conclusions, are contrary to general experience. It is generally held that a sewage-contaminated water yields a higher free than albuminoid ammonia: that this is generally the case with sewage and with sewage effluents is beyond doubt. The fate of these ammonias and their relation to each other must surely depend upon the bacterial content, the degree of aeration and so forth, of the soil through which the water passes. What happens in stoppered bottles is unlikely to be a certain guide to what happens during percolation through a soil. Again, the author's observations with regard to the effect of decaying vegetable matter are, I think, contrary to other published experiments.

The whole question is one of bacterial action, as is, indeed, observed by the author at the end of his paper.

In series M. and O. the sudden disappearance of the nitrites after the 63rd day, following on the gradual rise, is remarkable. Possibly the exhaustion of available oxygen is the cause of this. Is this likely to happen under natural conditions?

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* Criticisms made, either wholly or in part, subsequently to the meeting, are given in the first person.—EDITOR.

The slowness of the evolution of the free ammonia from water D. in Table V. is also remarkable. Such a result is not in accordance with that usually found on distilling effluents. I suggest that the later fractions are not free ammonia at all, properly so called, but are the result of the gradual hydrolysis of some nitrogenous constituent.

The author is rather sweeping in his references to data other than the ammonias, nitrites and nitrates. Has the chlorine content been justly discredited? Surely in certain circumstances the chlorine affords most valuable indications. Similarly "oxygen absorbed" figures, likewise discredited by the author, are often of value. Is it correct to say that, as an invariable rule, nitrates have no significance in chalk waters? They may be extremely significant.

Is it not the case that to suggest a general standard by which potable waters can be judged is, in any case, a futility? A standard of 0·05 parts of "nitrates" per 100,000 would condemn the bulk of the water supplies of this country, unless the water were in that privileged class in which "nitrates are of no significance."

In classing certain views which are widely held and, one had thought, well established, as "fallacies based upon isolated analyses of sewage and sewage effluents," the author lays himself open to the retort that his own conclusions are based upon the results of the examination of two particular contaminations in one particular water under one particular set of conditions—and those not natural ones.

Mr. O. HEHNER: This paper is written with the almost total disregard of the immense mass of work which has been done during the last half century on the analysis of potable waters and the interpretation of the analytical results. If anything stands out, it is the clearly recognised fact that no single estimation by itself is of any value, but that all the figures obtained by a somewhat comprehensive examination must be taken together in order that a fair judgment may be possible. It is futile to say, as does Mr. Frederick, that the chlorine content has been discredited, and that the "oxygen absorbed from permanganate" is useless, or that "albuminoid ammonia" has no significance. The interpretation of a water analysis may be likened to the reading of a book; each single word or sentence means nothing; it is the total which counts. This total in all cases should include chlorine, sulphuric acid, nitric acid, nitrite, phosphoric acid, free ammonia, albuminoid ammonia, oxygen absorbed, total solids, hardness, and, wherever possible, a bacterioscopic examination for, at least, *B. Coli*. An excess of chlorine may come from the sea; of sulphates from deposit of gypsum; of nitrates (within limits) from the chalk; of free ammonia from reduction of nitratet through iron pipes, or a spongy iron filter; of albuminoid ammonia from peas or other harmless vegetable matters, and so on. Then the origin of the water must, wherever possible, be taken into account, and the sample be compared with the natural unpolluted water from the same stratum or from the immediately surrounding district. An experienced water analyst can read from his results whether a water is polluted with house drainage, including urine, or with animal and manurial drainage, or with soakings from a churchyard, with brewery or similar washings, or is normal for peat water or river water; whether it comes from the chalk or sandstone, from near the sea, or the strata beneath the London clay, polluted or not polluted. Mr. Frederick would destroy all possibility of such judgment.

It may be noted that he re-discovers the fact, known for nearly 50 years, that urine does not give up its nitrogen, or only a small portion of it, in the form of free ammonia, whilst water polluted with fæcal matter in the natural way nearly always does.

Dr. J. C. **Thresh**: The results of the experiments detailed are interesting, but I fail to see how they assist in interpreting the results of analyses of natural waters. What would have been preferable would have been the study of some natural waters from sources which had been most minutely examined, and which were kept under observation for a considerable period. It seems to me to be safer to regard all waters as having at some period, however remote, been polluted, and to make such examinations thereof as will justify an opinion as to whether they now are "pure and wholesome" and "suitable for domestic purposes." These words, "pure and wholesome" and "fit for domestic purposes," never occur in the paper. Obviously Parliament, in insisting that potable waters should possess these properties, distinguished between pure and wholesome, and even knew that a pure and wholesome water might be unfit for domestic purposes. The analyst may decide about the purity, and even the fitness for domestic use, but the question of wholesomeness is a medical one, and does not depend on the free or albuminoid ammonia, the nitrites and nitrates, all of which, in the amounts likely to be found in a potable water, are absolutely harmless. I take it that a "pure" water is one which is free from colour, odour, taste, turbidity, free from lead or copper and excess of saline matter of an objectionable nature. The excess would also show whether the water was fit for such domestic purposes as washing and cleaning. The "wholesomeness" depends almost entirely on the bacterial contents—in fact, entirely on these in the absence of any constituent of a poisonous nature. The chemical character of a water can be determined without any attempt at estimating the constituents to the third and fourth place of decimals. The age is passing, if it has not already passed, when upon the chemical analysis alone depend all our conclusions regarding the purity and wholesomeness of water.

Mr. S. F. **Burford** said that experiments *in vitro* were not to be compared with results in the field. No careful men were basing information on water, nowadays, on nitrites, nitrates, and albuminoid ammonia only, for so much depended on the district from which the water came. In his opinion, the question of albuminoid ammonia had been much over-rated, and, within his experience, he remembered only three instances of contamination in which albuminoid ammonia was present. The amount of oxygen absorbed was another factor to be taken into consideration.

He considered that methods that were applicable to one class of water were not applicable to others, as, for instance, river water which contained no chlorine. He mentioned a district he knew where three totally different supplies of water were given to the public—one gave high ammonia, one gave chlorine, and one was highly calcareous water. When the question under consideration was merely the water supplied to one individual on his farm, the author's methods of analysis would be very satisfactory, but he considered the analyst must make a finer mesh, according to the purposes to which the water was to be put.

Mr. R. L. **Collett** pointed out that where the author had added only urine his nitrite determination was very steady, but where he had added fæces great changes were to be found in his results. Further, in the presence of copper or of chlorinated waters, he got very steady readings. In his opinion, the paper emphasised the importance of the bacteriological examination of water in conjunction with the chemical analysis.

Dr. **Bernard Dyer**: It has long been recognised that the water from a cesspool or drain percolating through but a few feet of subsoil into a well may have its nitrogen so completely oxidised by nitrifying organisms that the well-water, though heavily polluted, may yield but negligibly small traces of free and albuminoid ammonia, whilst its nitric nitrogen may be raised to even 1 part or more per 100,000. On the other hand, pure waters of deep well origin contain nitrates far in excess of the small figure which the author in one part of his paper suggests as evidence of objectionable impurity—a figure which would class the whole of the London water supply as "non-potable," and probably most of the supplies in the country, excluding the soft upland waters of Wales and the North. This, indeed, the author elsewhere seems to recognise when he makes the surely too sweeping assertion in the other direction, that in waters from certain strata nitrates have no significance.

Chemical analysis, while, if construed in the light of reason and experience, lending valuable aid in judging of the suitability of water for drinking purposes, is often of less assistance than investigation of the bacteriological condition of the water. This often enables the analyst to distinguish between what may be called "proximate" pollution and pollution so remote either in time or in space as to have become presumably innocuous.

REPLY BY MR. FREDERICK.

With regard to the courteous remarks of these authorities I must immediately point out that my standpoint has been fundamentally misconceived. I am considered to have examined a number of polluted artificially prepared samples, and on the results obtained to have founded a thesis on the interpretation of the results obtained in water examination practice. What I actually did was to make a number of experiments in an endeavour to obtain confirmation of opinions tentatively formed as a result of extended practical experience. That this is the correct standpoint is evident, beyond all question, from the opening paragraph of the paper.

It does not appear to be realised that the investigation was primarily directed towards determining the chemical changes which occur in samples during the time which elapses between collection and analysis.

There seems to be a general consensus of opinion that I have failed to realise the importance of the bacteriological examination of water. Even with only an elementary knowledge of the subject, one knows that this is, of course, an absolutely essential complementary examination, but in this paper I am dealing with the chemical aspect only, and specifically stated so. It could, with equal truth, have been pointed out that I did not make a microscopical examination of the sediment, or a spectroscopic examination of the dissolved solids. In point of fact, almost every one of the 1,000 or so samples of water on which this paper is founded was examined independently by a distinguished bacteriologist, and the fact that in approximately 99 per cent. of cases our opinions agreed, is the best proof that the principles on which I work and which I have enunciated in this paper are sound.

I am taken to task for stating that the value of the chlorine figure as an indicator of excretal pollution is discredited. In this paper it is shown that with comparatively gross pollution the chlorine is only increased 0·15 with urine and with fæces 0·007 parts per 100,000, and there does not seem any reason to revise my opinion.

Mr. O. Hehner states that I have disregarded the immense mass of work done during the last half century on the analysis of potable waters and the interpretation of the analytical results. If this work has been done, which I question, it has left little of permanent value, for the ideas of to-day seem much the same as those formed subsequent to the publication of Wanklyn's epoch-making work in 1867.

Dr. Dyer states that the limit for nitrates would class the whole London water supply as non-potable. The only water which can be considered definitely to be free from nitrates derived from strata, and to which therefore the limit is applicable, are collected rain waters and distilled waters; the London supply is obtained from rivers and wells.

The Crystalline Bromides of Linseed Oil.

BY HAROLD TOMS, M.SC. (LOND.), A.I.C.

(WORK DONE UNDER THE ANALYTICAL INVESTIGATION SCHEME.)

(*Read at the Meeting, December* 5, 1923.)

IT is well known that when an ethereal solution of linseed oil is treated with bromine an "insoluble" substance is obtained. When Hehner and Mitchell (ANALYST, 1898, **23**, 310) first drew attention to this, it was suggested that estimation of this compound might be used as the basis of a method for the evaluation of linseed oil. The "oil bromide test," however, has never been satisfactory, for, although many investigations (Ingle, *J. Soc. Chem. Ind.*, 1911, **30**, 344; Eibner, and Muggenthaler, *Chem. Zentralbl.*, 1913, **1**, 567; Sutcliffe, ANALYST, 1914, **39**, 28, 388; Gemmell, *ibid.*, 297; and Davidson, *J. Ind. Eng. Chem.*, 1921, **13**, 801 ANALYST, 1921, 466), have been undertaken with a view to placing it on a sound basis, no method has been evolved for preparing the compound in a chemically pure condition.

The oil bromide is somewhat insoluble in ordinary solvents, and this has prevented previous workers from obtaining it in a pure condition. Hehner and Mitchell (*loc. cit.*) purified their material by subjecting it to a long process of extraction, and in this way obtained a substance which melted at 143·5° to 144° C., and gave, on analysis, the following figures:

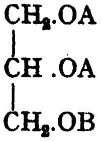
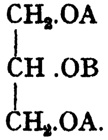
Found	C	= 32·97	Calculated for ash-free substance	C	= 33·29
	H	= 5·42		H	= 5·48
	Br	= 56·18		Br	= 56·74
	O	= 4·44		O	= 4·49
	Ash	= ·99			—
		100·00			100·00

Since $C_{57}H_{96}O_6Br_{14}$ requires C = 34·27; H = 4·81; Br = 56·11 and O = 4·81 per cent., it was suggested that the compound is a mixed bromo-glyceride of the composition

$$C_3H_5 \left\{ \begin{array}{l} C_{18}H_{29}O_2Br_6 \\ C_{18}H_{29}O_2Br_6 \\ C_{18}H_{33}O_2Br_2, \end{array} \right.$$

i.e. a glyceride containing one oleic acid and two linolenic acid radicles. More recently Davidson (*loc. cit.*) has pointed out that this composition corresponds equally well with that of linolenin-dilinolin bromide.

In passing, it may be noted that in neither paper is any mention made of the fact that any glyceride containing two molecules of one acid and one of another can exist in two isomeric forms. Thus:

(i)  and (ii) 

$$\text{(i)}\ \begin{array}{l} CH_2.OA \\ | \\ CH\ .OA \\ | \\ CH_2.OB \end{array} \quad \text{and (ii)}\ \begin{array}{l} CH_2.OA \\ | \\ CH\ .OB \\ | \\ CH_2.OA. \end{array}$$

It will be noted also that a substance having the constitution represented by 1 can exhibit optical isomerism.

In 1921 Davidson (*loc. cit.*) succeeded, with great difficulty, in crystallising some of the crude bromide from carbon tetrachloride. The product thus obtained melted at 151° C. (corr.), and contained 59·24 per cent. of bromine. This figure, as Davidson points out, approaches that required for dilinolenin-linolin bromide (59·42 per cent.). From other solvents he obtained small amounts of material giving bromine contents varying from 57·38 per cent. to 59·24 per cent. The melting points of these products are not stated. The same writer concludes that the crude oil bromide is mainly dilinolenin-linolin bromide mixed with a small amount of some other (unidentified) bromo-glyceride containing a smaller percentage of bromine.

The experiments recorded below were undertaken with the object of determining the nature of the precipitated "oil bromide," and, if possible, of finding a trustworthy method of testing drying oils by means of bromination.

PART I.

The following preparatory experiments were repeated several times in duplicate, first with a sample of oil of unknown origin, and then with Calcutta oil (iodine value 180·9), kindly given by Messrs. Blundell, Spence & Co., Ltd., of Hull. The products obtained in each case were identical in composition.

EXPERIMENTAL.

THE BROMINATION OF LINSEED OIL.—In a typical experiment 40 grms. of oil were dissolved in 300 c.c. of ether and 2 c.c. of glacial acetic acid were added. The solution was cooled by means of a stream of water and stirred mechanically while 16 c.c. of bromine (excess) were slowly run in. The stirring was continued for some hours and, after standing overnight, the supernatant liquor was decanted. Ether (100 c.c.) was added, and the mixture stirred vigorously for an hour; after standing, the supernatant liquor was again removed. This process was repeated twice more, but on the last occasion the mixture was filtered through an ordinary filter and left to drain for some hours. The residue was then boiled for several hours with alcohol and again filtered. This gave a white granular product (28 grms.) which, after drying on a porous tile, softened at 130° C. and melted at 140° to 145° C.

The ether residue left, after removing the solvent, a semi-solid sticky mass, which did not set after standing for several months. This has not been further investigated.

PURIFICATION OF THE "OIL BROMIDE."—With the object of finding a suitable crystallising medium, some 25 different solvents, mainly esters, were tried. Of these, ethyl acetate, ethyl formate, ethyl aceto-acetate and amyl acetate were the most successful. The first-named solvent gave the best results in that the product was obtained in a granular form, and could be readily separated by filtration; hence it was used in all subsequent experiments.

The crude "oil bromide" was boiled with ethyl acetate, the solution filtered hot and left to cool. After some hours the fine granular precipitate was collected on an ordinary filter, and when nearly dry was transferred to a flask, boiled with alcohol, and filtered hot. The residue (substance A) was then boiled with ether, filtered, and left to dry on a porous tile. The omission of this last operation usually resulted in the crystals caking together into a hard horny mass.

From 40 grms. of oil there were obtained 28 grms. of crude bromide (70 per cent.); this, on crystallisation, gave 7·5 grms. of pure product (18·7 per cent.). This corresponds with 11·2 per cent. of the unsaturated glyceride in the original oil.

From the ethyl acetate mother liquor there was obtained, on complete removal of the solvent, a large amount of a crystalline material (substance B), which in the crude state melted below 100° C.

SUBSTANCE A.—The substance which crystallises from ethyl acetate appears to be practically insoluble in this solvent in the cold. It melted at 153° C. (corr.), and further crystallisation failed to raise its melting point. A microscopical examination showed that it is microcrystalline, since it reacts to light through crossed Nicols. There were also indications of a biaxial structure.

On analysis the following results were obtained:

Found Br = 59·40 and 59·50; C = 31·4 and H = 4·18
Calc. Br = 59·42 ; C = 31·7 and H = 4·36.

This corresponds to linolic dilinolenic bromo-glyceride, $C_{57}H_{94}O_6Br_{16}$, which substance can exist either as the symmetrical or the optically active compound. Since neither linseed oil nor solutions of this compound show any rotation, it is put forward tentatively that this glyceride is either a racemic mixture, or more probably, the symmetrical compound.

Several determinations of the solubility of this pure bromide in ethyl acetate show that the figure lies between 0·15 and 0·20 grm. per 100 c.c. at temperatures ranging from 15 to 20° C.

SUBSTANCE B.—The residue obtained from the ethyl acetate mother liquor was dissolved in ethyl formate and decolorised by boiling with animal charcoal, and after many crystallisations from ethyl formate a small amount of material was obtained which melted at 117° C. (corr.) after washing with cold alcohol—an operation which prevented the substance from caking. This material reacts to light through crossed Nicols, and is undoubtedly crystalline, but no information regarding the structure of the crystals was obtainable.

On analysis the following results were obtained:

Found Br = 52·53 and 52·10 per cent.; C = 36·8 per cent.; H = 3·93 per cent.
Calc. Br = 52·23 per cent.; C = 37·2 per cent.; H = 5·33 per cent.

This corresponds approximately to $C_{57}H_{98}O_6Br_{12}$, which may be trilinolic bromo-glyceride or one of the several possible isomers of the mixed glyceride containing the three radicles oleyl, linolyl and linolenyl.

In the analysis of both substances it was found that the lime method of estimating the bromine (which was used by Hehner and Mitchell) gave low results, whereas the Carius method and a modification of the method of Stefanow (described below) gave the required theoretical results.

MODIFICATION OF THE METHOD OF STEFANOW.—The substance was decomposed with sodium ethylate, and the bromide precipitated as silver bromide in the usual way, with the use, however, of a known amount of 0·1 *N* silver nitrate solution. The excess on titration with ammonium thiocyanate always gave high results, so the silver bromide was collected on a Gooch crucible, and washed free from "fat" with ether. This gave consistent and theoretical results.

Attempts were made to remove the bromine from Substance A, but these were entirely unsuccessful. This is somewhat remarkable in view of the ease with which α- and β-linolenic acids (*cf.* Erdmann and Bedford, *Ber.*, 1909, **42**, 1324, 1334) are obtained by the hydrogenation of hexabromstearic acid.

Again, all attempts to hydrolyse the material failed completely; hence confirmation of the presence of linolic and linolenic acid residues could not be obtained. Up to the present all efforts to synthesise any of the glyceride containing the acid radicles have also been unfruitful.

PART II.

ESTIMATION OF THE CRYSTALLINE BROMIDE.—The comparative insolubility of the precipitated bromide in ethyl acetate suggested the idea that the weight of this compound obtainable might bear a definite relation to the drying value of the oil, and hence serve as a rapid means of evaluating linseed oil. Attempts were made, therefore, with several samples of oil, to correlate the percentage yield of bromide with the iodine value. The results obtained are shown below.

Sample number	I.	II.	III.	IV.*
Iodine value	180·9	179·5	184·8	183·3
SERIES I.				
Percentage of bromides..	19·26	(1) 18·26, (2) 18·50, (3) 18·20	19·15	15·80
$\frac{\text{Iodine value}}{\text{Percentage}}$	9·39	9·83	9·65	11·6
SERIES II.				
Percentage of bromides	24·31	21·93	25·29	23·26
$\frac{\text{Iodine value}}{\text{Percentage}}$	7·44	8·18	7·31	7·88
SERIES III.				
Percentage of bromides	22·10	20·10	21·30	19·49
$\frac{\text{Iodine value}}{\text{Percentage}}$	8·18	8·93	8·67	9·40

* Sample IV. was a dull yellow oil containing particles of solid matter which settled out as a sludge, very soluble in ethyl acetate.

In the first series about 1 c.c. of oil was weighed accurately and dissolved in 10 c.c. of ethyl acetate. Liquid bromine (1 c.c.) was then run in slowly, rise of temperature being avoided. After some time the precipitate was collected on a Gooch crucible, washed with ethyl acetate (20 c.c.), and dried at 80° C.

In the second series about 1 c.c. of the oil was weighed and dissolved in 10 c.c. of ethyl acetate. A slight excess of a solution of bromine in glacial acetic acid (1 c.c. =0·595 grm. Br.) was run in slowly to prevent rise of temperature. After standing for some time the liquid was decanted through a Gooch crucible, and the precipitate was washed with ethyl acetate (10 c.c.) and dried to constant weight at 80° C.

In the third series the estimations were carried out as in Series 2, except that the precipitate was washed, finally with alcohol.

Excluding Sample 4, the results obtained in the first series are somewhat more consistent than those in Series 2 and 3. It is interesting to note also that in Series 1 the melting point of the product was in each case about 148° C., whereas in the Series 2 the figure was about 140° to 141° C., and in Series 3, 142° to 144° C. The presence of glacial acetic acid seems therefore to increase the yield of precipitate (*cf.* Davidson, *loc. cit.*), but renders the product less pure. By washing with alcohol some of the impurity is removed, as is shown by the decreasing yield and the rise in melting point.

SUMMARY.

Two definite crystalline compounds have been isolated from linseed oil in the form of their bromides. The analyses indicate that they are (1) a linolic-dilinolenic bromo-glyceride, and (2) the trilinolic bromo-glyceride or an oleic-linolic-linolenic bromo-glyceride.

No synthetic confirmation of these results has yet been obtained. Data have been obtained which indicate that the amount of the most unsaturated glyceride in linseed oil is a constant proportion of the total unsaturated bodies present.

In conclusion, the writer wishes to thank Mr. C. Ainsworth Mitchell, who, with Mr. Otto Hehner, originally discovered the "oil bromide," for having unreservedly placed this investigation in his hands, and also the Society of Public Analysts for the grant which defrayed much of the expense incurred.

KING'S COLLEGE, UNIVERSITY OF LONDON,
STRAND, W.C.2.

DISCUSSION.

Mr. C. A. MITCHELL said that the author's work was an important contribution to our knowledge of the chemistry of linseed oil. Owing to the great variations in the results obtained in estimating the "insoluble bromide" of linseed oil, there had been a tendency to discard the method in favour of the estimation of the amount of linolenic hexabromide yielded by the fatty acids, but the drawback to this was the risk of oxidation of the fatty acids prior to bromination, so that the use of the oil itself, if the drawback mentioned could be overcome, was preferable. The author had now shown them the cause of these variations, which was that the "insoluble bromide" was a mixture of two relatively insoluble bromides, and that the proportion of these in the precipitate varied with the conditions, such as solvent used for washing the product, the length of time of purification, and so on. It should now be possible to obtain concordant results by estimating the more insoluble of the two bromides. He was not surprised to hear of the failure of the author to remove the bromine from these compounds. He had, himself, repeatedly tried to displace it by the action of nascent hydrogen continued for days at a time, but without success. Possibly the action of hydrogen in presence of a catalyst might be more effective.

Mr. MITCHELL added that Mr. Hehner had read Mr. Toms' paper, and wished to congratulate him upon the results he had obtained, and to express his regret that he was unable to be present to take part in the discussion.

Mr. TOMS, in reply, said that Dr. Gordon, of King's College, London University, (to whom he had sent the substances), was his authority for saying that the bromides were micro-crystalline; he had reported that they were crystalline and not amorphous—the one was biaxial, the other was of a different structure. With regard to obtaining the molecular weight by freezing, such a thing was obviously impossible, owing to the insolubility of the bromides.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

SPRING WATER OF UNUSUAL COMPOSITION.

RECENTLY we received a sample of water, which was taken from a spring in a wood in the vicinity of Glasgow, the object being to determine its suitability as a water supply. As it has an unusual composition, we give below a complete analysis:

	Parts per 100,000.
Aluminium sulphate	3·81
Ferrous sulphate	·01
Calcium sulphate	1·72
Magnesium sulphate	1·07
Magnesium chloride	·61
Sodium chloride	2·59
Sodium nitrate	·04
Silica	traces
Organic matter	·60
Total solid matter	10·45
Free ammonia	·005
Albuminoid ammonia	·007

There were neither carbonates nor free acid present, and the proportions of the various ingredients were calculated according to the form in which they were most likely to exist on evaporation. Although we have made hundreds of analyses of spring water, this is the only one that we have found to contain aluminium sulphate. We have had various samples of pit water containing large proportions of ferrous sulphate with smaller proportions of aluminium sulphate and free acid, but none giving a similar analysis to the above.

R. T. THOMSON.
JAMES SORLEY.

CITY ANALYST'S LABORATORY,
BATH STREET, GLASGOW.

THE VOLUMETRIC ESTIMATION OF VANADIUM IN STEEL.[*]

IT has been found that the green colour of chromium sulphate can be considerably reduced, or even destroyed, by a suitable addition of cobalt sulphate, since the two colours are complementary. The best ratio appears to be approximately 2 per cent. of chromium to 3 per cent. of cobalt, *i.e.* in a 1 per cent. chromium steel 1½ per cent. of cobalt would be added as sulphate. In fact, in this case, the solution can be made nearly colourless in a volume of 500 c.c. With 4 per cent. of chromium 6 per cent. of cobalt would be added as sulphate. In this case the colour is a pale green. With higher amounts the colours only partly compensate, and the pink colour of cobalt interferes with the end-points. The blank solution for colour comparison must be treated with the same amount of cobalt sulphate as in the test. The method has been found practicable up to 5 per cent. of chromium.

Conversely, a cobalt steel containing up to about 7 per cent. of cobalt can be treated with chromium sulphate so as to obtain the required ratio mentioned, and to eliminate the pink colour of the cobalt, before proceeding with the volumetric estimation.

A. F. ETHERIDGE.

RESEARCH DEPARTMENT,
WOOLWICH ARSENAL.

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[*] *Cf.* ANALYST, 1923, **48**, 588.

UNCOMMON ANIMAL FATS.

THE writer has personally obtained the fats of the Ceylon wild animals, the analysis of which is recorded below.

The Ceylon leopard, *Felis pardus*, is more often spoken of locally as the cheetah. The usual ground colour of the body is a yellowish brown, although sometimes it is very light, or even so dark as to merit the name of black leopard. In each case the skin is thickly studded with black rosettes with pale centres. A full-grown leopard will measure as much as seven feet from nose to tip of tail, and stand 23 to 25 inches high at the shoulder. It feeds on any kind of smaller animal, and will kill full-grown bulls.

The Sambhur, *Cervus unicolor*, is known in Ceylon as the elk. The usual colour of a buck is dark brown, and that of a female a greyish brown. The fur is very coarse and shaggy, so that the skin makes a poor trophy. A good buck stands from 13 to 14 hands in height at the shoulder, and will weigh as much as 600 lbs. The antlers are three tined. Irregular heads with extra abortive points are sometimes found, antlers with seven points having been seen.

The spotted deer, *Cervus axis*, is generally considered to be the most beautiful of all the deer tribe. The colour is a light fawn spotted with white. There is a dorsal stripe from the neck to the tip of the tail. The males stand from 34 to 38 inches high at the shoulder. They are gregarious, and are met with in herds up to 40 or 50.

The peafowl, *Pavo cristatus*, is found in the low country of Ceylon.

The particular species of pig found in Ceylon is *Sus cristatus*, the Indian wild boar. It has a short massive body and big head provided with fighting tusks. The colour of the skin is a dark slaty grey, with scanty bristles. It is naturally a vegetable feeder, but is by no means adverse to a meal of carrion.

The fats of the animals are all solids at the ordinary temperature (81° F.), with the exception of that of the peacock, which is a yellow oil. This last is highly valued by the local "vederalas" (native doctors) for use in cases of stiff joints or contracted tendons. The following analytical results were obtained:

FATS.

	Specific Gravity 99/15° C.	Butyro-refractometer reading at 40° C.	Acid value.	Saponification value.	Iodine value (Wijs).	Unsaponifiable matter. Per Cent.
Leopard	0·8592	49·5	1·78	201·1	62·2	0·33
Sambhur	0·8610	45·7	4·37	210·6	22·4	0·56
Spotted deer	0·8640	44·3	4·98	212·3	23·8	0·52
Peacock	0·8947	54·5	8·70	215·3	66·4	0·34
Pig	0·8851	52·0	11·12	216·4	44·8	0·40

MIXED FATTY ACIDS.

	Solidifying point. °C.	Butyro refractometer reading at 40° C.	Neutralisation value.	Iodine value (Wijs).
Leopard	39·2	38·9	210·3	60·9
Sambhur	48·2	31·6	213·1	21·4
Spotted deer	47·8	31·6	215·6	22·65
Peacock	36·4	46·9	216·6	66·7

Leopard's fat does not appear to have been examined before, but its constants closely resemble those of the tiger recorded by Lewkowitsch, who gives m. pt., of fatty acids, 37·5° C.; saponification value, 200·8; neutralisation value of fatty acids, 208·2; and iodine value, 57·7.

The analyses of the fats of the two members of the deer tribe, in spite of their tropical habitat, are very similar to those of European deer quoted in Lewkowitsch, who, for example, gives for the Chamois:—Solidifying point of the fatty acids, 51·5° C.; saponification value, 203·3; neutralisation value of fatty acids, 206·5; and iodine value, 25·0.

European wild boar fat, however, differs considerably from the Ceylon wild boar fat; for example, the saponification value is given as 195·5 and the iodine value as 81.

WILLIAM NORMAN RAE.

CHEMICAL LABORATORY,
CEYLON UNIVERSITY COLLEGE, COLOMBO

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE THIRD QUARTER, 1923.

DURING the 3rd Quarter of the year, 1256 samples were submitted for analysis, of which 1087 were analysed under the Food and Drugs Acts, and 134 for the water department. Of the Food and Drugs samples 1049 were bought informally, and of these 43 were adulterated; 38 were bought under the provisions of the Acts, and of these 7 were adulterated.

MILK.—Of the 539 samples examined 38 were adulterated with water or were deficient in fat, and of these 38 no fewer than 33 contained less than 3 per cent. of fat, which is an unusually large proportion. Thirty-eight samples from 23 different farmers contained less than 11·5 per cent. of total solids.

CREAM.—Four of the 11 samples of cream were adulterated, as they contained from 0·23 to 0·35 per cent. of boric acid. None of them had a declaratory label and one was marked "Thick Rich Cream," although it was preserved cream. Two of the informal samples sold as preserved cream were free from boric acid, and should have been sold as cream.

VINEGAR.—Twenty-six informal samples were genuine, but one contained 3·0 per cent. of acetic acid, and one subsequent formal sample contained 2·9 per cent. Proceedings were taken, and the defendant, who undertook to sell from smaller casks in future, was ordered to pay the costs of the prosecution. (*cf* ANALYST, 1923, **48,** 544).

ZINC OINTMENT.—One of 5 informal samples contained 17·3 per cent of zinc oxide, instead of 15 per cent., the proper amount.

BORIC ACID POWDER.—One informal sample contained 80 parts of lead per million, being more than three times the limit of the B.P., and the vendor was cautioned. Nine other samples were genuine, containing from 4 to 24 parts of lead per million.

J. F. LIVERSEEGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

SALE OF A BUTTER MIXTURE.

JUDGMENT was given by the Justiciary Appeal Court at Edinburgh, on December 15, 1923, in a stated case in which a judgment by Sheriff-Substitute W. J. Robertson, of the County of Ayr, was submitted for review.

The proved facts were that J. and J. McKay, grocers, of Dalry, had sold to an inspector 1 lb. of margarine, in reply to a demand to be supplied with a certain butter mixture exposed in their window and labelled "Butter mixture, 1s. 2d. per lb." The analysis had shown the sample to be margarine containing less than 10 per cent. of butter fat.

The respondent had been charged with a breach of the Sale of Food and Drugs Acts for the sale of the sample, and also of the Margarine Act, in that the paper wrapper had not the word "margarine" upon it.

The Sheriff-Substitute had acquitted the respondent on the ground that the inspector asked for a particular article and received it, and that any misdescription was the misdescription of the buyer, and not of the seller. He had also acquitted him on the second charge, since the butter mixture was not delivered to the purchaser in any wrapper, and the fact that he had no wrapper in his shop was not relevant to the charge.

The Court, by a majority of two to one, upheld the appeal of the Procurator-Fiscal, and remitted the case to the Sheriff-Substitute with instructions to proceed.

Lord Cullen thought that the Sheriff was not entitled from the facts proved to infer that the purchaser's demand for "1 lb. of that butter in the window" was not truly a demand for butter.

Lord Sands considered that the merchant was guilty of a contravention of the Act, if, when asked for "that butter," he supplied a customer with a mixture resembling butter, without satisfying himself that the customer knew that it was not butter.

The Lord Justice-General pointed out that the Sheriff-Substitute had reported that it had been proved that, at the time of the purchase, the inspector had knowledge of the terms of the ticket. The exposure of the article for sale was an offer of a mixture not of any single article, nor of an article of any single "nature, substance and quality." The respondent was probably committing an offence under the Margarine Acts by selling, otherwise than as margarine, a compound resembling butter, but he was not charged with an offence under those Acts, but under the Food and Drugs Acts, for selling "butter" which was not of the nature, substance and quality of butter. It did not seem just or reasonable to hold, on the facts proved, that the purchaser's demand for "that butter" was a demand for "butter," in view of the fact that the purchaser was aware that what he was asking for was not "butter," but a "butter mixture." His Lordship was, therefore, of opinion that the Sheriff-Substitute was justified in acquitting the respondent.

Department of Scientific and Industrial Research.

FOOD INVESTIGATION BOARD.

RED DISCOLORATION (SO-CALLED "PINK") ON DRIED SALTED FISH.*

THE state known as "pink" is one to which dried, salted fish is liable.

The report begins with a full history of the subject, from which it appears that "pink" was first investigated in 1878. Much has since been written on the state, and the views as to its cause and hygienic significance are divergent.

The conditions necessary for the appearance of the condition are a temperature high for our latitude, and moist air. Hence "pink" with us is practically limited to the hottest months of our summer, but it is said that consignments of salt fish leaving us for the Mediterranean countries in an apparently sound condition not infrequently become badly affected with "pink" when they come into warm climates.

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* Special Report, No. 18. By P. C. Cloake, M.D., D.P.H. Pp. 22. 1923. H.M. Stationery Office. Price 1s. net.

Owing to the water-attracting qualities of the salt used in curing, moisture condenses in the cracks on the surfaces of salt fish, and between the apposed surfaces of fish when they are stacked. It is there, especially in the surface cracks, that "pink" begins.

In the process of drying and salting, the fish are handled freely and exposed to the dust from the air. Therefore the surfaces are, as might be expected, generally infected. The bulk of the organisms present, however, have nothing to do with "pink." They are for the most part the ordinary common forms of bacteria and exert no undesirable action.

"Pink" is due to at least two organisms—a "red coccus," which occurs in well-defined coccus and sarcinal growths, and an organism of a striking type, so far unidentified, to which the name organism X might for convenience be given. An alga is also present in great abundance on the surface of dried and salted fish. This occurs in uncoloured areas, as well as in the patches of "pink." It is therefore not held to be responsible, even in part, for the discoloration.

The "pink"-forming organisms, the red coccus and organism X, are remarkable in that they grow only in the presence of a large quantity of salt—over 15 per cent. They belong, therefore, to the group of organisms called, from their liking for salt, halophilic. Both have the further peculiarity that they grow slowly and need a moderately high temperature. At 77° F. it is about three weeks before visible colonies appear. Growth was found to be much more rapid at 99° F.

Organism X was recognised with difficulty, owing to the fact that it is destroyed by the procedure ordinarily adopted by a bacteriologist, who begins by dispersing his suspected substance in dilute solution of salt. This completely destroys X, since it breaks up in dilute solutions of salt into an amorphous slime. It was found, however, to preserve its character and to remain alive in strong brine. No signs of the spirochæte which earlier writers had described as a cause of "pink" were found.

The experiments described in the report seem conclusively to prove that "pink" is due to the use of salt, and more particularly solar salt, contaminated with these "pink"-forming organisms. The salt, as used by curers, becomes contaminated with them, as they are present on the salt-encrusted walls of stores, and on the surfaces of tables used by the operatives. The method of prevention is therefore simple, namely, to sterilise the salt, the utensils, and the rooms. Whether it is commercially feasible only those who know the magnitude of the losses which "pink" entails upon the industry can say.

Salt can be sterilised by heating for thirty minutes to 248° F.—a lower degree of heat will not suffice. Salt can also be sterilised, so far as these "pink"-producing organisms are concerned, by adding to it 5 per cent. of sodium bisulphite, but this is not to be recommended for hygienic reasons.

There is still another possibility, namely, drying the fish further than is done at present. Fish dried so that little moisture remains seems incapable of growing "pink." Unfortunately, as is shown in an appendix by Dr. Moran and Mr. Piqué on the absorption of water by salted and dried cod, the capacity for reabsorbing water, so as to become again palatable, is lost when drying is excessive. The appendix throws light upon the irreversible nature of the drying process. Curves are given which show that the rate of drying is also an important factor. The pieces of fish most rapidly dried showed the greatest capacity for reabsorbing water.

The Government Laboratory, Bangkok, Siam.

FIRST REPORT FROM NOVEMBER, 1917, TO MARCH 31, 1922.

THE Director of the Government Laboratory at Bangkok, Mr. A. Marcan, F.I.C., has issued his first report on the work of the year ending in March 31, 1922.

In November, 1917, the Department of Commerce took over the chemical work then being carried out at the Royal Mint, with the object of establishing a central laboratory for the whole country. The work outgrowing the accommodation available at the Mint, a new building was erected on a central site, and was occupied in September, 1919, and all Government Departments were invited to submit their chemical work, and analyses were made for the public on payment of fees.

This laboratory is fitted with modern equipment, including coils, chilled by ice, connected with the main water supply, electric heating and power, and exhaust fans for ventilation. During the period under review 26,140 analyses were made, consisting principally of coinage silver and bronze for the Mint.

CHEMICO-LEGAL WORK.—This included toxicological analyses of drugs, foods, stomach contents, etc., and the examination of stains for blood. The materials chiefly used for poisoning are plants which are of common occurrence throughout the country.

Much work is needed to obtain accurate knowledge of the poisonous active principles of some of the indigenous plants, and the Laboratory would be grateful to anyone who could give authoritative information as to some of these plants and their properties.

One narcotic drug, a species of *Dictyophora*, is not known to have been reported elsewhere. It is dried and burnt, and the fumes blown on to the sleeping victim, with the result that a sound sleep is produced and theft can be committed in safety.

In the period reviewed 16 cases were submitted, involving the analysis of human viscera, and 83 miscellaneous materials suspected to contain poison. Positive results were obtained in 33 per cent. of the cases submitted.

Atropine, one of the active principles of *Datura* species, was detected in 12 of the cases, arsenic in 8, corrosive acids in 3, mercury in 2, arsenic with nicotine in 2, strychnine, opium, atropine with morphine, chloroform, *Gloriosa superba*, and sulphur (for producing suffocating fumes) in one case each.

POISONOUS PLANTS IN SIAM.—The following is a preliminary list of the plants known to contain powerful or poisonous principles. Three are known to have been used, and others are likely to be used, for criminal purposes:—*Abrus precatorius*, Linn. (seeds); *Allamanda cathartica*, Linn. (juice and leaves); *Nerium odorum*, Soland (root and bark); *Cerbera odollam*, Gaerta (seeds); *Thevetia neriifolia*, Juss. (juice); *Calotropis gigantea*, Br. (juice); *Datura*, Spp. (seeds); *Euphorbia*, Spp. (juice); *Jatropha curcas*, Linn. (seeds); *Croton tiglium* (seeds); *Excoecaria agallocha*, Linn. (juice); *Cannabis indica*, Linn. (dried plant); *Antiaris toxicaria*, Lesch. (juice); *Gloriosa superba*, Linn. (rhizome); and *Dictyophora*, Sp. (dried plant burned). Imported: *Strychnos nux vomica* beans.

OTHER WORK.—This included 57 cases of counterfeit coining, 38 samples of drugs for the Government Medical Depot, 54 drugs under the Morphine and Cocaine Act, and 29 samples of suspected illicit opium, for which a rapid and accurate method of dialysis was devised.

The mixed ethyl esters of chaulmoogra oil were prepared from native oil, and were used with satisfactory results in the treatment of leprosy, and extract of rice (vitamin extract) was made on a small scale for use in cases of beri-beri. Provision has now been made for the preparation of these extracts on a technical scale, and also for the establishment of a laboratory for the examination of natural products, such as oil seeds, tanning materials, drugs, etc., on a semi-technical scale.

Ministry of Health.

The following circular has been sent to the clerks of Authorities administering the Food and Drugs Acts:

SALE OF FOOD AND DRUGS ACTS.

Public Health (Milk and Cream) Regulations, 1912 and 1917.*

CIRCULAR 462.

Sir,

1. I am directed by the Minister of Health to request that a copy of the report of the Public Analyst for the fourth quarter of the present year may be transmitted to this Department during the month of January, together with a copy of a report by the Medical Officer of Health upon the administration of the Public Health (Milk and Cream) Regulations for the year 1923. In this connection I am again to draw attention to Memorandum 36/Foods, a copy of which was forwarded to the Council in January, 1921, and to ask that the reports may be prepared on the lines indicated in that memorandum.

2. In view of the increasing practice of selling milk in sealed bottles it is desirable that the attention of sampling officers should be drawn to the difficulty which is sometimes found in dividing a sample of bottled milk so as to secure the uniform distribution of the milk fat throughout the three parts of the sample. Where the cream has risen freely in the bottle or where some of it adheres to the sides it may be found useful to decant the milk into a larger vessel and to return a small quantity in order to rinse out the bottle before the final mixing and division are effected.

3. The Minister desires to recommend to the consideration of the Council the following suggestions which he has received from the Society of Public Analysts with regard to the sampling of prescribed medicines:—

"(i) That the inspector be instructed, prior to dividing the sample into three parts, to mark, in the presence of the vendor, the height the contents reach in the bottle in which the medicine is originally supplied to him by the vendor. That the bottle so marked be submitted to the Analyst in order to enable him to determine the total quantity of medicine supplied.

"(ii) That the Analyst and the Medical Officer of Health be both consulted as to the type of prescription it is desirable to use for the purpose of checking the accuracy of dispensing, and that, in the event of any substantial inaccuracies being disclosed by analysis, both these officers be consulted as to the desirability of instituting proceedings."

The object of the first suggestion is to enable the Analyst to ascertain the total quantity of each of the ingredients (including the water or other "vehicle") present in the medicine. If he receives only a portion of the sample, representing an unknown fraction of the whole, he is only able to ascertain the relative percentages of the several ingredients; but if he is also provided with information as to the total quantity of the medicine dispensed he can then deduce the total amount of each of the ingredients as supplied by the Chemist. These particulars will obviously be of importance to the Council in considering what action they should take in regard to a medicine which has been inaccurately dispensed.

The Minister believes that the second suggestion is in accordance with the practice which is already largely adopted by Local Authorities. It is clearly desirable that, where a sample of medicine is taken for the purpose of checking the accuracy of dispensing, it should be so chosen as to be capable of accurate analysis. It is also desirable in considering whether or not proceedings should be taken under the Sale of Food and Drugs Acts that proper regard should be paid both to the degree of accuracy obtainable in the analysis of the article in question and to the margin of error allowable in ordinary dispensing operations.

I am, sir, your obedient servant,

R. B. Cross, *Assistant Secretary.*

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* With regard to the dispensing of medicines, somewhat strangely included in these Regulations, *see* Analyst, 1923, **48**, 492.—Editor.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Proteins of Wheat Bran. D. B. Jones and C. E. F. Gersdorff. (*J. Biol. Chem.*, 1923, **58**, 117–131.)—From wheat bran, by which is meant the outer seed coats together with the under-lying layer of cells which contains the protein, three proteins, *viz.*, an albumin, a globulin and an alcohol-soluble protein have been isolated. The bran, which was exceptionally clean, was rapidly washed in cold water and dried at a low temperature to remove the adhering particles of other portions of the wheat kernel and ground to a powder. This contained 17·25 per cent. of protein (N × 6·25). By successive exhaustive extractions with distilled water, 4 per cent sodium chloride solution, boiling 70 per cent. alcohol and 0·5 per cent. sodium hydroxide solution, 86·61 per cent. of the total protein in the bran was extracted (N × 6·25). The preparations and properties of the three proteins isolated are described. Their percentages, expressed in terms of the total protein in the bran, were as follows:—Albumin, 16·64; Globulin, 13·62; Alcohol-soluble protein, 31·01 per cent. Analyses showed them to have the following average elementary percentage composition:—Albumin: N, 15·42; C, 53·21; H, 6·71; S, 1·35. Globulin: N, 17·76; C, 53·43; H, 7·40; S, 0·91. Alcohol-soluble protein: N, 15·35; C, 54·25; H, 6·75; S, 1·35. P. H. P.

Estimation of Reducing Sugars by means of Cupro-potassium Solution· L. Maquenne. (*Bull. Soc. Chim.*, 1923, **33**, 1681–1692).—The method here considered (*cf. Bull. Soc. Chim.*, 1898, **19**, 926; *Compt. rend.*, 1915, **161**, 617; 1916, **162**, 145, 207, 277) consists essentially in estimating by means of potassium iodide and sodium thiosulphate in an acid medium and, without separation of the cuprous oxide, the copper remaining in solution in the cupro-potassium solution reduced by Haen's method. The criticisms advanced against this method by Fleury and Boutot (*Bull. Soc. Chim. Biol.*, 1922, **4**, 361) are refuted, and it is shown that the method gives results as accurate as those obtained by the procedure recommended by these authors, and is to be preferred for various reasons. T. H. P.

Clarification of Solutions in the Analysis of Sugar Products. L. Eynon and J. H. Lane. (*J. Soc. Chem. Ind.*, 1923, 463T.)—The authors have previously found that the estimation of sugars by Fehling's solution is vitiated by the presence of calcium, and that this can be overcome by the use of potassium oxalate instead of sodium sulphate to remove the lead. This was not known when the experiments were carried out on which is based the view that normal lead acetate, unlike the basic acetate, does not precipitate invert sugar from solutions of raw sugar-products, although it does appear to remove a small proportion of reducing non-sugars. The authors have accordingly re-investigated these points in the light of their previous discovery.

They find that in the clarification of solutions of raw sugar-products no reducing sugar is co-precipitated by the normal lead acetate in the presence of either tannic acid, egg albumin, or the organic non-sugars of cane molasses and beet molasses. (These were specially prepared by suspending the washed lead precipitate in water and passing through it sulphuretted hydrogen, filtering from the lead sulphide and boiling off the excess of gas from the filtrate.) They also show that no reducing sugar is co-precipitated by precipitation of the lead by means of potassium oxalate. They are unable to confirm the experience of A. H. Bryan who states that if a solution of dextrose, magnesium sulphate and ammonium tartrate is treated with lead acetate and then with potassium oxalate, 0·9 per cent. of the dextrose is removed. The estimation of sugar-products without preliminary clarification involves two errors, that due to reducing non-sugars precipitable by lead and that due to the presence of calcium salts. The nett effect of these errors in the case of raw sugars is generally negligible, but may be serious in the case of sugar-syrups and molasses. The following procedure is therefore recommended for low grade products:—The sample (12·5 grms.) is dissolved in water, treated with 25 c.c. of a 10 per cent. solution of normal lead acetate and with a little alumina cream, if necessary, and made up to 250 c.c., the whole being thoroughly shaken and filtered. One hundred c.c. of the filtrate are treated with 10 c.c. of a 10 per cent. solution of potassium oxalate, made up to 500 c.c., shaken and filtered. This one per cent. solution of the sample is used for the estimation of invert sugar and sucrose, as under (*a*) and (*b*), respectively. (*a*) The solution is titrated as previously described (*ibid.*, 1923, 32T.) (*b*) One hundred c.c. of the solution are treated with 15 c.c. of normal hydrochloric acid, diluted to about 150 c.c., heated to boiling and boiled for two minutes. The solution is then cooled, neutralised with sodium hydroxide, diluted to 200 c.c., and titrated as under (*a*). R. F. I.

Application of Miscibility to the Detection of Adulteration of Cacao Butter and to the Identification of Various Pure Products. H. Rosset, Marange and Vinter. (*Ann. Falsificat.*, 1923, **16**, 454–468.)—Two simple forms of apparatus are described for determining low and high miscibility temperatures. With a suitable liquid, such as pure aniline, which may be identified by its miscibility curve with a well-defined liquid like 50 per cent. aqueous alcohol (1) pure cacao butters yield virtually identical miscibility curves, the extreme differences of temperature being 2·7° C.; (2) with the exception of illipé butter, the common adulterants of cacao butters, do not exhibit the phenomenon of miscibility; and (3) the presence of minimum proportions of such adulterants in cacao butter may be detected by means of the temperatures of reciprocal miscibility.

The fat or chocolate to be examined is extracted with ether, preferably anhydrous, in a Soxhlet extractor, the solution obtained being concentrated by distillation at the ordinary pressure and then evaporated at 50° C. under 12 mm. pressure for 4 to 5 hours, to eliminate moisture and ether; fatty matters, simply melted and filtered, may be used directly without appreciable error. The results of measurements of the miscibility of aniline with various samples of pure and adulterated cacao butters, etc., are given.

The presence of small proportions of nitrobenzene in aniline and of benzene in chlorobenzene may be detected similarly by investigating the miscibility with aqueous alcohol. T. H. P.

Detection of Saccharin and Dulcin in Vinegar and Products containing Acetic Acid. G. Reif. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 217–223.)—A new German food law limits the amount of dulcin in foodstuffs to 0·2 grm. per litre or per kilo. The following method is advocated for the detection of dulcin and saccharin:—One hundred c.c. of the liquid, or 100 c.c. of extract in the case of a solid substance, are made just alkaline with sodium bicarbonate and shaken out for half an hour with two successive quantities of 100 c.c. of ether. The ether extract is distilled, the residue dissolved in hot water in a test tube, a few drops of mercuric nitrate solution added, and the mixture warmed for a few minutes; in the presence of dulcin a violet colour is produced which is darkened by the addition of lead peroxide. The alkaline liquid is now acidified with sulphuric acid and extracted with ether. Then the ether extract is washed with a little water, evaporated, and tested for saccharin as usual. If the substance contains benzoic acid or much fat it should be first extracted with about 10 c.c. of ether before being made alkaline for the dulcin test. Should salicylic acid be present, it must be eliminated by oxidation with alkaline permanganate. The acid ether extract is therefore evaporated at a low temperature, and the residue dissolved in weak alkali, warmed, and potassium permanganate added in slight excess; the mixture is now acidified and excess of permanganate is discharged by the cautious addition of sulphurous acid. The clear solution is next extracted thrice with ether, and the residue, after evaporation, converted into salicylic acid by fusion at about 250° C. in a silver crucible with sodium hydroxide, after which it may be identified by means of ferric chloride in the usual way. H. E. C.

Polarimetric Estimation of Malic Acid. F. Auerbach and D. Kruger. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 97–154.)—The greatly enhanced optical activity of malic and tartaric acids in combination with uranium or molybdenum affords an accurate method for their estimation, either alone or in the presence of each other (*cf.* ANALYST, 1923, **48**, 346). Essential conditions are the presence of an excess of the activating metal and a suitable P_H value, which is secured by the addition of a suitable buffer substance. The molecular rotations are to some extent dependent on concentration, and have the following values at about 0·1 mol/litre for the sodium flame; temperature 18° to 20° C. uranium tartrate, +650°; uranium malate, −700°; molybdenum tartrate, +1044°; molybdenum malate, +1020°. For the estimation of malic acid the solution is exactly neutralised with sodium hydroxide, 0·1 mol/litre of di-sodium citrate added, the mixture shaken for about four hours with 3 grms. of uranyl acetate in 25 c.c. of the solution, and the rotation then observed. If molybdenum is to be used, the concentration of malic acid should not be less than 0·02 mol/litre; sodium acetate 2·5 mgrm. mol., or 1 c.c. of glacial acetic acid and 0·4 grm. atoms per litre of molybdenum as ammonium molybdate, are added to 25 c.c., and the rotation is observed as before. In the case of tartaric acid the solution, which should contain between 0·1 and 0·15 mol/litre, is neutralised and treated, as before, with uranium acetate and sodium citrate solution: if the solution is of less strength than 0·1 mol. per litre the molybdenum method may be used by adding glacial acetic acid and ammonium molybdate in the proportions already quoted. When both acids are present in the same solution advantage is taken of the fact that there is a change of sign in the rotation of malic acid with uranium and molybdenum. The rotation is therefore observed after treating portions of the solution with uranium acetate and ammonium molybdate respectively, and the proportions of the two acids calculated with the help of graphs which are given in the text. The presence of other organic acids, or of considerable quantities of alkali salts, does not materially affect the rotation of uranium malate or of molybdenum tartrate, but interferes with that of the other two compounds, so that the process is not applicable directly to malic and tartaric acids together in the presence of such substances. The method may be applied to the estimation of both acids in fruit juices or extracts by the precipitation of the proteins, pectins, etc., then separation of the organic acids by means of their barium salts. The rotation of the barium salts on the addition of uranium or molybdenum is given, and details of the application and the calculations are also shown. H. E. C.

Estimation of Malic Acid in Fruit Products. F. Auerbach and D. Krüger. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 177–217.)—The practical application of the barium malate method of separation of malic acid and its polarimetric estimation by means of uranium and molybdenum salts is described in detail (*cf.* preceding abstract). For the estimation of malic acid in fruit juices, jams and similar products, the alkalinity of the ash is first determined, then slightly more than the required quantity of hydrochloric acid is added to 25 c.c. of the extract, and the pectins precipitated by diluting the liquid to 100 c.c. with alcohol. After filtration, 75 c.c. are neutralised by the addition of barium carbonate, then 2 c.c. of a saturated solution of barium acetate are added, the barium malate is precipitated by diluting the liquid to 250 c.c. with alcohol, warming, and allowing the mixture to stand overnight. The barium malate precipitate is filtered off, washed with alcohol, dried and brushed into a 50 c.c. flask, and to it are added 0·05 grm. of barium tartrate, 0·1 grm. of barium citrate, and water to the mark. The solution is shaken for several hours, filtered, and the rotation of the filtrate observed after treatment with uranium and molybdenum as follows:—(*a*) To 20 c.c. 2·5 mgrms. mol. of disodium citrate and 3·5 grms. of uranyl acetate are added, and the mixture shaken for four hours and made up to 25 c.c. (*b*) To 10 c.c. are added 10 c.c. of a saturated solution of ammonium molybdate and 2 c.c. of acetic acid, then the mixture is allowed to stand in the dark for three hours and diluted to 25 c.c. If the solutions are coloured, they may be decolorised with animal charcoal as usual. The malic acid content is calculated from the observed rotations in the manner already described (*loc. cit.*). Corrections are given for the volume of the precipitates. Dextrin, sucrose, dextrose and fructose are not precipitated with the barium malate in the above process, and do not interfere unless present in large proportions. Preservatives, such as formic, benzoic or salicylic acid, do not affect the accuracy of the method. The following results were obtained with fruit juices:

	Per Cent.		Per Cent.
Pear juice	0·32	Apple juice	0·60–0·73
Cherry juice	0·56–0·67	Red current juice	0·052
Gooseberry juice	0·088	Raspberry juice	trace
Bilberry juice	0·044	Cranberry juice	none
Juniperberry juice	0·013	Tomato juice	0·013

H. E. C.

Mustard Flour and Oil in the Wine Industry. J. C. Delage. (*Ann. Falsificat.*, 1923, **16**, 483–491.)—Ground black mustard, either untreated or after being washed with 5 times its weight of water, exhibits marked antiseptic properties, and is also able to destroy unpleasant flavours. Thus, a quantity of the mustard containing 0·0014 grm. of allyl mustard oil is sufficient to render a litre of wine aseptic. This effect is not only far greater than that of sulphurous acid, but also more permanent, as yeasts do not develop tolerance towards the mustard oil. In white wines the ground mustard produces a slight turbidity which renders filtration of the wine necessary, so that the mustard oil itself should be used in such cases. The oil is best employed in a 3 per cent. solution in 50 per cent. aqueous alcohol. T. H. P.

Iodimetric Estimation of Thiosinamine. Morvillez and R. Meesemaecker. (*J. Pharm. Chim.*, 1923, **28**, 442–445.)—An aqueous potassium iodide solution of iodine is decolorised by a solution of thiosinamine, and the estimation is carried out by acidifying 10 c.c. of the thiosinamine solution in an Erlenmeyer flask with sulphuric acid, adding 20 c.c. of 0·1 *N* iodine solution, and leaving the mixture in the dark for 15 minutes. The non-combined iodine is then estimated by titration with 0·05 *N* sodium thiosulphate solution in the presence of starch, as indicator, and 12 c.c. of chloroform. If A is the number of c.c. of thiosulphate solution required, the strength of the thiosinamine solution is found from the formula:—$(A - n/2) \times 0{\cdot}0058 \times 10$. As a result of experiments with different lengths of reaction time (5 to 90 minutes) in neutral and acid solutions, it was found that the best conditions are 15 minutes in acid solution. The results obtained by this method are in close agreement with those by the Charpentier-Volhard and Denigès methods of estimation, which both depend on the decomposition of silver nitrate by the thiosinamine with the formation of silver sulphide, and the present method effects a great saving of time. The method is also applicable to the estimation of volatile mustard oil in essential oil of mustard. D. G. H.

Autoxidation of Chloroform. A. M. Clover. (*J. Amer. Chem. Soc.*, **1923, 45,** 3133–3138.)—Chloroform undergoes oxidation in diffused light, with the formation of a peroxide, which is probably dichloro-carbon-peroxide. This substance is formed at a gradually increasing rate, and its accumulation proceeds up to a certain point, where rapid decomposition sets in. Beyond this point the oxidation of the chloroform continues, although very little peroxide is found in the solution. The phosgene, carbon dioxide, chlorine and hydrochloric acid present in the oxidised product are derived from the decomposition and hydrolysis of the peroxide. The presence of 0·1 per cent. of alcohol retards the autoxidation, and in a test experiment with that amount only a trace of peroxide was found after 6 weeks. With 0·05 per cent. of alcohol autoxidation was only prevented for about a week. Phenol benzyl alcohol, petroleum spirit, and purified paraffin oil have also been found effective preservatives. The preservative acts as an anti-catalyst.

Eucalyptus Oils as Germicides. A. R. Penfold and R. Grant. (*J. Roy. Soc., N.S. Wales*, **1923, 57,** 10; *Chem. Trade J.*, **1924, 74,** 10.)—One per cent. suspensions of crude eucalyptus oils and their pure constituents in 7·5 per cent. resin soap solution were tested by the Rideal-Walker method. Of the ten commercial eucalyptus oils examined, that of *E. radiata* had the strongest germicidal value, its coefficient being 10 to 12; its active principle was piperitol. The pure active principles of the oils gave the following Rideal-Walker coefficients:—Australol, 22·5; geraniol, 21; citral, 19·5; and piperitol, 13. It was found that a concentrated preparation diluted with water gives a lower coefficient than an original dilute preparation of the same strength, probably owing to dilution disturbing the emulsion in the case of a concentrated suspension.

Biochemical, Bacteriological, etc.

Physiological Assay of Insulin Preparations. H. Penau and H. Simonnet. (*J. Pharm. Chim.*, 1923, **28,** 385–395.)—After investigating various methods of examination which have been put forward the authors eliminated all chemical methods, having found physiological methods, particularly those in which rabbits and dogs are used, to be the most reliable. Since the physiological response of individual animals varies somewhat, a minimum of four animals should be used in all experiments and the physiological unit for insulin is defined, according to the Toronto workers, as the quantity of active principle which, injected into rabbits weighing about 2 kilos. and fasting for 16–24 hours, depresses the blood sugar from 0·11 per cent. to about 0·045 per cent. The clinical unit is taken as a third of this. Objectively, the hypoglycæmia is accompanied by characteristic convulsions which, however severe, can be cured in a few minutes by intraperitoneal injection of dextrose, the dose being to a certain extent proportional to the amount of insulin injected.

Since the physiological and therapeutic values of a given insulin preparation are not always identical (which may be accounted for by regarding insulin as a

polyvalent complex of which the various parts have different pharmacodynamic properties), the effect of various preparations on depancreated dogs was tried. The authors conclude that the pharmacodynamic activity of insulin is best investigated by the Canadian rabbit method, supplemented by the effect produced on the sugar content of the blood and urine of depancreated dogs—an action perhaps more comparable with that which occurs in the case of the human patient.
D. G. H.

Aqueous Extracts of Pancreas. II. New Facts. H. A. Piper, R. S. Allen and J. R. Murlin. (*J. Biol. Chem.*, 1923, **58,** 321–336.)—Insulin is not a protein. This refers to the product obtained from the pancreas of the ox and pig. A subsequent paper will give the method of preparing it protein-free. It has been obtained as a white or greyish-white amorphous powder. The insulin molecule, if it be a chemical entity, must be large, for it withstands dialysis in thin vegetable parchment for many hours; thus it can be separated from diffusible salts. Insulin is only with difficulty separated from protein, since it is adsorbed by this class of substance. The hypoglycæmic action of insulin varies with different conditions. There is an apparent development of potency. Where the first injection sometimes gives no reaction an injection from the same solution the next day yields a satisfactory test. The potency is not reduced when the filtrate, after standing, shows a growth of bacteria, and remains even when a hydrogen sulphide odour is apparent. The development of the hypoglycæmic reaction in rabbits has much to do with the exact reaction to which the neutralisation is carried after extraction in 0·2 *N* hydrochloric acid. Insulin is fairly stable. Practically protein-free, it has been kept for 4 months. The lowest acidity at which it has been observed to keep well is $P_H = 5{\cdot}7$. To obtain large yields the range of acidity between $P_H = 4{\cdot}3$ and 5·7 at any stage just preceding filtration should be avoided. Insulin is not a concentrated form of glutathione.
P. H. P.

Aqueous Extracts of Pancreas. III. Precipitation Reactions. C. P. Kimball and J. R. Murlin. (*J. Biol. Chem.*, 1923, **58,** 337–346.)—Rapid preparation of insulin for medicinal purposes requires that it should be removed from solution by some method more expeditious than that of drying. In this attempt to find a method of precipitation the preparations used were as protein-free as possible; many reagents which are well known protein precipitants failed to give any precipitate. A specific amount of the reagent to be tested was added to the preparation, and the precipitate was thrown down by centrifugal action, taken up in distilled water and tested as soon as possible on rabbits. The supernatant liquid was dried, with or without dialysis, depending upon the nature of the additive, and similarly dissolved and tested. Tables of the results are given. Positive reagents were:—Ammonium sulphate, sodium chloride, trichloroacetic acid, acetone and methyl, ethyl, isopropyl, *n*-butyl, isoamyl and caprylic alcohols. Probably insulin, when pure, is insoluble in neutral water. It gives no protein reactions and the most potent preparations that have been analysed have had a low nitrogen content, 4 to 6 per cent. dry weight.
P. H. P.

Oxidation of Dextrose by means of Iodine in Presence of Insulin. G. A. Alles and H. M. Winegarden. (*J. Biol. Chem.*, 1923, **58**, 225–234.)—This investigation was carried out to discover whether insulin, alone or in the presence of certain animal fluids, has any influence upon dextrose *in vitro*. It was known before, and is confirmed here, that the rate of oxidation of various sugars by iodine in solutions of sodium bicarbonate or sodium phosphate varies greatly with the nature of the sugar; thus a study of the relative rates of oxidation of dextrose, before and after treatment with insulin, should give a sensitive means of finding whether any of the dextrose has been transformed by it into any other substance, even into a stereomeric hexose. Experiments, which are described, were made with dextrose alone, with mixtures of dextrose and insulin, with insulin and liver extract, and with insulin and blood serum or oxalated blood. Tables of results are given. In no case was any difference detected in the rate at which the iodine is consumed. Therefore no appreciable reaction takes place between dextrose and insulin, even in the presence of the animal fluids mentioned; thus the metabolic process must be more complicated in character; also, there is little promise of developing a method of assay for insulin on the basis of its action on dextrose outside the body. P. H. P.

Fat-Soluble Vitamins. XIV. H. Steenbock, E. B. Hart, J. H. Jones and A. Black. (*J. Biol. Chem.*, 1923, **58**, 59–70.)—Additional justification is given for speaking of the fat-soluble vitamins rather than of a fat-soluble vitamin, as support, by experiments carried out upon dogs and chickens, is lent to the idea that the anti-rachitic vitamin is an entity distinct from vitamin *A*, the anti-ophthalmic substance. Pups were weaned and fed on a standard rickets-producing diet, whereby the inorganic phosphate and calcium of the blood were reduced. By the administration to 3 dogs of 1 c.c., 4 c.c. and 12 c.c. daily respectively of cod liver oil, freed from vitamin *A* by aeration, the blood phosphorus and calcium were restored to normal, and the ash content of the bones was increased. One dog was kept as a control. The cod liver oil was not able to correct growth interference; vitamin *A* was probably needed. Some butter fat, in addition to its ration of cod liver oil, caused the first dog to become normal. The dog receiving 4 c.c. of cod liver oil per day developed a purulent rhinitis, and the one having 12 c.c. a keratitis, thus furnishing additional proof that the cod liver oil was free from vitamin *A*. The aerated cod liver oil increased the inorganic phosphates and calcium of the blood of chickens and re-initiated growth, but it must not be inferred that the birds do not need vitamin *A*, since they were put upon the experimental ration at an advanced age and weight and had had opportunity to store this vitamin. P. H. P.

Fat-Soluble Vitamins. XV. R. M. Bethke, H. Steenbock and M. T. Nelson. (*J. Biol. Chem.*, 1923, **58**, 71–103.)—Experiments are described by means of which the inorganic relations in the bloods and bones of rats when fed on various deficient diets were studied. Poor calcification of bone occurred in rats on a synthetic ration of purified food constituents deficient in fat-soluble vitamins, even with the addition of 15 c.c. of skimmed milk or 0·5 c.c. of whole milk per rat per day. Normal calcification occurred with 20 c.c. of skimmed milk or 1 c.c. of whole milk. Data were accumulated on the variation in composition of blood and bone with age in the rat, preliminary to further studies. The ash content of the femurs and humeri totalled 66 per cent., a maximum value, when the animals weighed 375 to 425 grms. The blood phosphates and calcium were slightly higher in the very young than in the older animals. Additions of calcium to a basal ration caused a similar increase of growth to that caused by additions of fat-soluble vitamins, as found in cod liver oil, thus indicating the existence of a quantitative relation between vitamin and calcium in effecting the assimilation of the latter. Probably the beneficial results from the feeding with calcium salts are due to the mass action of the calcium counteracting the effects of a vitamin deficiency. Absence of normal growth tended to give a reduction of the calcium content of the blood and ash content of the bones. The phosphorus of the blood was more constant, but was depressed as the calcium increased. With casein as the protein, given in the proportion of 18 per cent. of the ration, the phosphorus requirements of the rat appeared to be complied with. Increased growth did not result from the addition of a neutral mixture of sodium and potassium phosphates. This was reflected in the composition of the blood and bones. The calcium of the blood was consistently depressed where no fat-soluble vitamins were added, the greatest depression being where the most phosphate was added. Radiations with ultra-violet light for 10 minutes daily in the absence of fat-soluble vitamins in the diet brought up both the calcium and phosphorus of the blood. P. H. P.

Presence of Vitamin *A* in Frozen Pork. A. M. Wright. (*J. Soc. Chem. Ind.*, 1923, **42**, 509T.)—Following work on the presence of vitamin *B* in Frozen Flesh Food (ANALYST, 1923, **48**, 611), the author has investigated a point which has long been a matter of dispute, namely, the presence of vitamin *A* in lard. The experimental work is on the same lines as in the former investigation, and the diet given to the cockerels consisted of pork containing 25 per cent. of fat which had been in cold storage for 9 years, starch (with or without butter fat for control purposes), the same salt mixture as before, and the juice of swede turnips. The following results were obtained:

	Controls A. B. & C Vitamin present. Grams.	Controls Vitamin A deficient. Grams.	Pork with butter fat. Grams.	Pork: no butter fat added. Grams.	Pork; butter fat first 28 days only Grams.
Original weight	161	164	179	169	168
7–10 days ...	165	166	195	201	197
10–14 ,, ...	177	173	206	225	213
21 ,, ...	214	161 (died)	241	268	258
28 ,, ...	247	—	277	279	290
35 ,, ...	300	—	279	296	302
42 ,, ...	302	—	295	300	315
49 ,, ...	321	—	346	338	342
60 ,, ...	333	—	350	347	360

It is evident, therefore, that even after 9 years in cold storage at a temperature varying between 2° and 15° F. vitamin *A* is present and remains active in pork fat.
D. G. H.

Differentiation of Vitamin *A* from the Anti-rachitic Factor. E. Lesné and M. Vagliano. (*Comptes rend.*, 1923, **177**, 711–712.)—The results of experiments on the administration of cod liver oil to rats subjected to an anti-rachitic diet confirms the non-identity of the anti-rachitic factor of the oil with the fat-soluble vitamin *A*. The latter may be introduced into the organism in any way, but the former is active only when ingested. T. H. P.

Preservation of Blood for Analysis. F. V. Sander. (*J. Biol. Chem.*, 1923, **58**, 1–15.)—The data experimentally obtained show that samples of human blood can be preserved for 5 to 6 days, and that the values for non-protein nitrogen, urea, uric acid, creatinine, creatine and sugar will have the same clinical significance as those values obtained by an immediate analysis of the blood. The urea, uric acid, creatinine, creatine and sugar content of blood can be kept constant for at least 2 weeks, but the non-protein nitrogen value slightly increases, and this cannot be accounted for by an increase in ammonia and amino-acid content. A mixture of 0·01 grm. of sodium fluoride and 0·001 grm. of thymol for each c.c. of blood is recommended for the preservation of blood for 6 to 14 days, and, with the use of this, the blood need not be drawn under sterile conditions, except with regard to the patient. Sodium fluoride or thymol alone will not preserve the blood constituents investigated for a period of 5 to 6 days, but the above mixture is apparently very efficient. Both have but little effect upon the reagents used in each analysis and cause no interference with methods. Higher concentrations of the preserving materials are not necessary for preservation, and do not upset the analysis, but cause hæmolysis to take place in much less time. No single preserving substance can be relied upon to maintain unchanged the values for non-protein nitrogen, urea, uric acid and sugar for a period of 48 hours; creatinine and creatine remain constant for several days in unpreserved blood and in blood containing many of the common preservatives. P. H. P.

Identification of Blood Meal of Mosquitoes. C. G. Bull and W. V. King. (*Amer. J. Hygiene*, 1923, **3**, 491–496.)—The blood meals of mosquitoes can be identified easily by the precipitin test if the specimen is collected within an hour after the mosquitoes have fed, and fairly well up to 12 hours afterwards, but specimens collected up to 24 hours afterwards usually give negative or non-specific reactions. Identification is possible 36 hours afterwards if the fed mosquitoes are kept in an ice box. Blood in an advanced stage of digestion fails to react with any of the precipitin sera. The sera preparations, the collection of the blood specimens, and the test are described in detail. Specimens collected as described may be kept for 3 or 4 months before being tested, since they undergo no further disintegration. Known specimens were obtained as controls by allowing mosquitoes to feed on man and different domestic animals. Control tests reproduced show the weakest point in the method to be the failure of reactions with the anti-human serum. A highly potent anti-human serum could not be obtained. P. H. P.

Qualitative Tests for Acetone Bodies. E. J. Bigwood and W. S. Ladd. (*J. Biol. Chem.*, 1923, **58**, 347-361.)—The authors made both quantitative and qualitative analyses on the same samples of diabetic urine and carried out experiments to throw light upon the selectivity, sensitivity and quantitative significance of these tests. The following is the technique used. *Sodium nitroprusside test.*—Add 20 drops of a mixture of 10 c.c. of glacial acetic acid and 10 c.c. of a 10 per cent. sodium nitroprusside solution to 10 c.c. of urine in a test-tube, shake, and layer on this 1 to 2 c.c. of concentrated ammonium hydroxide solution. At the contact of the fluids a purple ring appears. *Ferric chloride test.*—To 10 c.c. of urine 10 c.c. of a 10 per cent. ferric chloride solution are added, but more if necessary to clear the precipitate. A brown colour appears. Readings are taken at intervals with both these tests as the colours deepen. It was found that a pure acetone solution and a solution of diacetic acid free from acetone each gives a colour reaction with sodium nitroprusside. Only the latter solution gives a colour reaction with ferric chloride. Electrolytes present in urine, especially sodium chloride, tend to intensify the colour of the ring in the sodium nitroprusside test. Quantitatively the tests serve only as crude approximations in indicating the amounts of acetone and diacetic acid present. Of the two, the ferric chloride test appears to give less eccentric results. P. H. P.

Anaerobes from Water Samples. P. D. Meader and E. A. Bliss. (*Amer. J. Hygiene*, 1923, **3**, 394-400.)—The presence of gas in broth containing 1 per cent. of lactose is regarded in the *Standard Methods of Water Analysis* of the American Public Health Association (1920) as a presumptive test for the presence of *B. coli* in the water under examination. Sometimes gas is found when *B. coli* cannot be isolated. The origin of this gas is important. There appears to be a variety of organisms, morphologically unlike *B. coli*, which may be a source of error and difficulty in water analysis, because of gas production in lactose broth. Anaerobes have been isolated from 16 to 21 per cent. of 76 tubes of lactose broth, inoculated with samples of water. These tubes showed gas production, but no *B. coli* could be isolated. Of 25 strains of anaerobic bacilli tested, 2 fermented lactose, with evolution of gas. Anaerobic bacilli were isolated from raw water and from water subjected to various methods of purification, but the presence of these organisms in water appeared to bear no relation to the aerobic count. Anaerobic organisms appear to have been an unimportant factor in the fermentation of lactose in the samples investigated. P. H. P.

Survival of Bacteria in Flies. R. W. Glaser. (*Amer. J. Hygiene*, **1923**, **3**, 469-480.)—The biting stable fly, *Stomoxys calcitrans*, the cattle horn fly, *Lyperosia irritans*, and the house fly were used in this work, the last for purposes of comparison, since it would be unsafe to compare the results obtained on the first two with the published data on *M. domestica*, owing to the region in which the work was done. The experiments are described. Results showed that the intestine of a house fly at any age is always a veritable reservoir for bacteria. *Stomoxys* harbour bacteria within them, but the number is much smaller. New bacteria are not present in the daily food of *Stomoxys*, as is the case with the omniverous house fly, for the former feeds on sterile vertebrate blood, which may have an inhibiting effect on the bacteria already inside the fly. Recently emerged horn flies contain very few bacteria, and adults practically none. The practical sterility of the adult intestine is strange, since the larvæ live in cow dung and ingest quantities of bacteria. Probably the bacteria are inhibited and destroyed as the fly is transformed and assumes adult life. P. H. P.

Moulds on Frozen Meat. A. M. Wright. (*J. Soc. Chem. Ind.*, 1923, 488T.) —Several workers have proved that the moulds they have found on frozen meat have included *Mucor mucedo*, a *Rhizopus*, *Penicillium glaucum*, *Thamnidium elegans*, and *Cladesporium herbarum*, and that in several cases of "black spot" one or other of these (except *Thamnidium*) has been found to be the cause. Brown spots have been caused by a saccharomyces which grew at −2° C., but not at −9° C. *Cladesporium herbarum* has been found to produce "black spot" on meat kept at −5·5° C., but in New Zealand this appears to be the least common cause. The author has found that if *Mucor mucedo* has been grown on meat at from −2° to −1° C. till well established and then placed in cold storage at −12° to −15° C., a black spot has developed. Similar results have been obtained with *Penicillium glaucum*, but the initial temperatures were about 4° C. The author therefore concludes that no one species of fungus is responsible for "black spot" on frozen meat. In every case that the author has investigated he has shown that at some time during the cold storage the temperature has risen above 0° C., as a result of defective insulation, and, in some cases, sufficiently high and for a sufficiently long period to allow actual putrefaction to take place. It has been said that prolonged cold storage in itself is the responsible factor in producing moulds and "black spot." The author, however, can produce mould on cold-stored meat in one month; on the other hand, he has found meat free from moulds after nine years' cold storage under strict conditions of temperature and sound insulation. Two cases came before his notice of a feathery growth being diagnosed by an authority as "mould," which were subsequently proved to be not mould at all. One consisted of crystals of salts and organic compounds which separate from meat juices during freezing, and the other was composed of the cotton fibre from the bagging which had adhered to the carcase as fluff. The temperature in the cold storage of meat should never rise above −9° C. (*Cf.* ANALYST, 1923, **48**, 549.) R. F. I.

Toxicological and Forensic.

A Case of Poisoning by Sodium Arsenate. M. Bridel. (*J. Pharm. Chim.*, 1923, **28**, 395–397.)—The maximum dose of sodium arsenate given by the French Codex is 1 cgrm. at a time and 2 cgrms. in 24 hours, but the toxic dose for a man is 0·252 to 0·263 grm., this being based on Kohn Abrest's figure of 0·115 to 0·120 grm. of arsenic acid. If prescribed in a solution containing 0·1 grm. in 300 c.c., 5 mgrms. in a dose and 1 cgrm. in 24 hours is generally ordered. Notes of a case are given in which a woman took 4 spoonfuls at 24 hour intervals of a solution containing 0·261 grm. per spoonful, *i.e.* 1·044 grm. of sodium arsenate in 72 hours. After the third dose headache, puffiness of the face and sore throat supervened, together with jaundice. Diarrhœa and vomiting followed, but only lasted two or three days, and gradual recovery took place. D. G. H.

The Swedish Commission on Arsenical Poisoning. Petrén. (*Acta Med. Scand.*, 1923, **58,** *Fasc.* II.–III; *Lancet*, 1923, **205,** 531–532.)—A summary is given of the work of the Commission of chemists and doctors appointed by the Swedish Government to investigate the questions of domestic poisoning by wall-paper, clothing, etc., due to arsenic. It was found necessary to fix certain standards for this class of poisoning. Thus, for arsenical house poisoning to be diagnosed, it must, among other things, be proved that the symptoms began within 1 to 3 months of exposure, and ceased within 3 months of the cessation of such exposure to house poisoning. When these criteria were applied, very few of the 91 cases reported to the Commission were authenticated.

Differences in Poisoning by Arsenic from Different Sources.—When arsenic is taken in large doses by the mouth, melanosis and polyneuritis are prominent sequelæ, whereas melanosis has never, and polyneuritis has rarely, been observed in cases of house poisoning. On the other hand, workers in arsenic, in contact with Schweinfürth's green, are liable to suffer from cutaneous lesions, which are practically unknown in cases of poisoning with arsenic given by the mouth. In the case of house poisoning, volatile compounds of arsenic appear to be responsible for the symptoms. The chemical investigations of Prof. Ramberg, a member of the Commission, have shown that the composition of the arsenical gases developed by the action of certain moulds is inconstant, and do not support Biginelli's hypothesis that the arsenical gas generated under these conditions is diethylarsine.

Other Findings of the Commission.—Much stress is laid on the great differences in the reaction of the body to the same compound. In the case of salvarsan, for instance, the reaction of the body would seem to depend to a certain extent on the nature of the pre-existing infection, and the reaction of syphylitics to salvarsan is probably peculiar to this class of case. In the case of influenza patients, however, the first and only toxic effect of salvarsan was vomiting. Confirmation has been obtained of the observations of Tinel and Leroid, who found that, whilst arsenic cannot reach the cerebro-spinal fluid through healthy membranes, it may do so when these membranes show morbid changes.

Agricultural Analysis.

Soil-point Method for Directly Estimating the Water-supplying Power of a Soil in the Field. F. Hardy. (*J. Agric. Sci.*, 1923, **13,** 355–360.)—The method is designed to show the actual water-supplying power of a soil to plant roots *in situ*; this power bears no relation to the total moisture content, and is largely dependent on the nature of the contact between the soil and the experimental point. One hundred ordinary hard graphite pencils (Hardtmuth's 4H.) are sharpened in a rotary mechanical sharpener, and the points then blunted by rubbing on sand paper; they are separated into 20 marked sets of 5, each set is weighed and at once placed in a celluloid case. Ten of the sets are planted out in a transverse across the field, the pencils of each set being inserted at right angles to the transverse. The planting is done by pressing a stout nail into the soil and inserting the pencil to a depth of 5 inches into the hole thus made. The planting is timed so that the points may be in contact with the soil for three hours, at the end of which time the sets are withdrawn, immediately replaced in their cases, and weighed as soon as possible. The experiment should be made in duplicate. The water-supplying power is expressed in hundredths of a grm. absorbed by one soil-point; soils on which sugar-canes grew vigorously had water-supplying power between 2·7 and 4·7; normal growth coincides with values between 2·7 and 2·3, and soils below 2 showed distinct wilting. H. E. C.

Effect of Movement of Soil Salts on the Standardisation Values of Electrodes used in Moisture Estimations. T. Deighton. (*J. Agric. Sci.*, **1923, 13,** 440–446.)—Electrodes were buried at various depths in a large mass of soil kept on a constant-level water table and protected from rain so that the moisture content at any depth varied only with the temperature and humidity of the atmosphere. Under these conditions movements of the soil and salts would be indicated by altered resistances. After making allowance for observed differences of temperature, the results show that the humidity of the atmosphere is without effect on the moisture content at depths greater than 3 inches. After several months water was added by means of a fine spray equivalent to 1 mm. rainfall, this had no effect on the resistance, but increasing amounts of water caused a lowering of the resistance. It is concluded that standardisation values in normal soils are not materially affected by showers. H. E. C.

Cause of Error in the Jodlbauer Process for the Estimation of Total Nitrogen. J. Bordas. (*Comptes rend.*, 1923, **177,** 696–697.)—Estimation of the total nitrogen in a fertiliser containing leather by Jodlbauer's modification of Kjeldahl's method gives low results, loss of nitrogen being caused by the reducing action of the tannin on the nitrate. Similar errors are encountered if pyrogallol, hydroquinone or pyrocatechol is present, but the non-reducing resorcinol is without effect. Hence when both tannin and nitrate are present, the total nitrogen must be estimated either by the Dumas method, or by Salle's or Desvarda's method (reduction in an alkaline medium), followed by Kjeldahl treatment of the residue of the distillation. This modification must be applied also to vegetable matter containing both tannin and nitrates. T. H, P.

Water Analysis.

Estimation of Dissolved Air in Small Quantities of Water. H. G. Becker and W. E. Abbott. (*J. Soc. Chem. Ind.*, 1923, 484T.)—On dissolving potassium hydroxide in tap water a vigorous evolution of dissolved air can be seen, and this observation is the basis of the method described. Twenty to thirty c.c. of the water to be tested are introduced into a simple special apparatus of which a sketch is given, allowed to dissolve the potash and the small bubble of air evolved is measured, passed through pyrogallol and again measured all in the same apparatus. As the potash would be expensive if the method were used regularly, a cheaper alternative was looked for. After several trials, choice was made of ammonium sulphate in the form of specially prepared air-free pellets. The methods of Letts and Blake or of Winkler are designed for use with 250 c.c. of the water, and are not accurate for quantities under 50 c.c. R. F. I.

Organic Analysis.

Detection of Halogen in Organic Compounds. J. Piccard and F. de Montmollin. (*Helv. Chem. Acta*, 1923, 6, 1020.)—In the case of volatile compounds, the flame test on copper wire is modified by introducing into the flame, 1 cm. above the substance, a small piece of copper wire gauze, which is attacked by the halogen acid. If the substance is rich in carbon and renders the flame luminous, it is held in the small flame of a Bunsen burner by means of copper wire; the combustion gases are made to traverse the flame of a second burner, inclined above the small flame, where the green coloration becomes visible. A sensitive reaction for the detection of small quantities of halogen in volatile liquids (*e.g.* benzene) is carried out in a vertical combustion tube, the upper end of which holds a filter paper impregnated with the liquid. The lower end of the tube is drawn out and bent upwards. The vapour issuing from the narrow tube is ignited, and a tiny piece of copper gauze held in the flame; the green coloration will develop in the colourless burner flame held over the small flame. W. R. S.

Estimation of Fluorine in Organic Compounds. J. Piccard and C. Buffat. (*Helv. Chim. Acta*, 1923, 6, 1047–1048.)—The substance is heated with potassium to about 400° C. *in vacuo*. From 0·1 to 0·2 grm. (*e.g.* phenyl fluoride) is sealed in a small thin-walled glass bulb so that the bulb is almost completely filled. The bulb is transferred to a pressure tube together with 20 c.c. of absolute ether and 0·5 grm. of potassium. The tube is drawn out to a capillary and evacuated completely, after which the capillary is closed. The bulb is then broken by shaking, and the tube heated some time over a free flame, with shaking, to expose fresh surfaces of the fused metal. For the completion of the reaction, the tube is heated in a tube furnace to about 400° C. After cooling, the tube is opened, and its contents taken up with alcohol and with water, any insoluble matter (carbon) being filtered off. The authors use conductometric titration with calcium chloride for the estimation of the fluoride in the solution. W. R. S.

Benzidine as a Reagent for Aldehydes. P. N. van Eck. (*Pharm. Weekblad.*, 1923, **60**, 1204–1208.)—Benzidine gives distinctive colour reactions with groups of aldehydes and with individual aldehydes dissolved in glacial acetic acid. For example, it gives colorations ranging from yellow to orange with aliphatic aldehydes; with benzaldehyde it forms a crystaline yellow deposit; with anisaldehyde, orange crystals; and with phenylacraldehyde, blood-red crystals. It affords a delicate test for vanillin, with which it gives an orange-red coloration, changing to red on diluting the solution with water.

Composition and Constitution of Elæostearic Acid. L. Maquenne. (*Bull. Soc. Chim.*, 1923, **33**, 1654–1655.)—Vercruysse's conclusion that the structural formula of elæostearic acid contains two multiple linkings (*Bull. Soc. Chim. Belg.*, 1923, **32**, 151) was also arrived at by the author (*Compt. rend.*, 1902, **135**, 696), who showed that this acid is extremely liable to undergo oxidation, and that it is both acetylenic and ethylenic in character. The melting point, 48° C., is confirmed for the acid. T. H. P.

Estimation of the Iodine Value of Oils and Fats by means of Pyridine Sulphate-bromide. K. W. Rosenmund and W. Kuhnhenn. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 154–159.)—Pyridine-sulphate-bromide ($C_5H_5N.H_2SO_4.Br_2$) is recommended for the iodine absorption test, as it forms additive compounds with oils without any substitution or oxidation. The reagent is prepared of 0·1 *N* strength by dissolving 8 grms. of pyridine and 10 grms. of sulphuric acid in 20 c.c. of glacial acetic acid (kept cool meanwhile), adding 8 grms. of bromine dissolved in another 20 c.c., and diluting the mixture to 1 litre with glacial acetic acid. The solution keeps well, but if stored in a large flask frequently opened in warm weather it should be periodically standardised. The oil or fat is dissolved in 10 c.c. of chloroform, a small excess of the pyridine-sulphate-bromide solution is added, and the mixture allowed to stand for 3 to 5 minutes. After this time the excess is determined either by adding potassium iodide solution and titrating the liberated iodine with thiosulphate, or by direct titration with standard arsenite solution. Tables of results show that the iodine value so estimated is strictly comparable with that given by the more usual iodine tri-bromide method. With the latter the value obtained is to some extent dependent on the excess added, but with the proposed reagent quite a small excess is sufficient and a large one is not deleterious. The reagent prepared in the above manner is also suitable for the estimation of arsenious acid; the acidified arsenic solution is coloured red with methyl orange, and the standard pyridine solution run in until the methyl orange is oxidised and its colour destroyed. Phenol may be estimated as tribromphenol by adding excess of the reagent and titrating back with arsenite solution. H. E. C.

Analysis of Soap Powder. F. F. Flanders and A. D. Truitt. (*J. Ind. Eng. Chem.*, 1923, **15**, 1232.)—The following method is recommended for the rapid estimation of soap and sodium carbonate in soap powders. Two grms. of the sample are dissolved in 100 c.c. of hot water, and the solution is titrated with 0·5 N hydrochloric acid, with the use of methyl-red as indicator; 2 c.c. of 0·5 N hydrochloric acid are then added, the mixture is boiled for ten minutes to expel carbon dioxide, and the excess of acid is titrated with 0·1 N sodium hydroxide solution. A further quantity of 2 c.c. of 0·5 N hydrochloric acid is added, the mixture is cooled, and extracted with three successive quantities of 25 c.c. of neutral chloroform; the chloroform extracts are filtered, the filter washed with 25 c.c. of chloroform, the filtrate boiled to expel carbon dioxide, and the hot solution titrated with 0·1 N sodium ethylate solution, phenolphthalein being used as indicator.

$$Na_2CO_3 = \frac{(\text{cc. } 0{\cdot}5\,N\ \text{HCl}) - (\text{cc. } 0{\cdot}1\,N\ \text{NaOH}) - (\text{cc. } 0{\cdot}1\,N\ C_2H_5ONa)}{2} \times 0{\cdot}0053.$$

$$\text{Anhydrous soap} = \frac{(\text{cc. } 0{\cdot}1\,N\ C_2H_5ONa)}{2} \times 0{\cdot}0306.$$

W. P. S.

Lubricant and Asphaltic Hydrocarbons in Petroleum. C. F. Mabery. (*J. Ind. Eng. Chem.*, 1923, **15**, 1233–1238.)—The author has investigated the nature of the petroleum hydrocarbons which cannot be distilled without undergoing decomposition; the crude oil was first distilled under 30 mm. pressure up to 300° C., and the undistilled residue subjected to fractional solution in a hot mixture of ether and alcohol. The specific gravity, molecular weight, and percentage composition of each fraction were then determined. The method was applied to petroleums from W. Virginia, Pennsylvania, Ohio, Texas and Russia. A comparison of the results shows that there is a difference between the lubricant and asphaltic hydrocarbons, and the higher specific gravity of the Texas and Russian lubricant hydro-carbons is due to their structure. Determinations of the iodine values show that the ring form of unsaturation applies only to the lubricant hydrocarbons, and these do not appear to enter into the formalite reaction of the Marcusson method. W. P. S.

Chemistry of Wood. Relation between Methoxyl and Lignin in Wood. G. J. Ritter. (*J. Ind. Eng. Chem.*, 1923, **15**, 1264–1266.)—The lignins isolated from soft and hard woods differ in composition, since a larger percentage (80 to 90) of the total methoxyl content of the wood can be recovered from soft wood lignin than from hard wood lignin (62 to 80 per cent. recovery). When the woods are heated with dilute alkali solution under pressure they yield the same percentage (approximately 63) of methoxyl in the form of methyl alcohol, but the composition of the residues differs; the hard wood residue contains the remaining methoxyl, whilst the soft wood residue contains none. This fact also suggests a different methoxyl linkage in the two lignins. W. P. S.

Estimation of Pentoses and Pentosans. II. Estimation of Furfuraldehyde. N. C. Pervier and R. A. Gortner. (*J. Ind. Eng. Chem.*, 1923, **15**, 1255–1262.)—Titration with potassium bromate solution, in the presence of hydrochloric acid and potassium bromide, is recommended for the estimation of furfuraldehyde in the distillate obtained when a pentose or pentosan is distilled with hydrochloric acid (*cf.* ANALYST, 1924, **49**, 47). Five c.c. of 20 per cent. potassium bromide solution are added for every 100 c.c. of the furfuraldehyde solution (distillate) and the acidity is adjusted to about 4 per cent. by weight of HCl by the addition of acid or alkali, as required. The mixture is stirred and 0·1 *N* potassium bromate solution is added from a burette at such a rate that the production of a yellow colour throughout the solution is avoided. When the end-point is approached, the bromate is added in quantities of 0·2 c.c., and the time required for the disappearance of the free bromine is determined electrometrically. A rather large increase in the time required is noted at the end-point of the titration, and the observations are carried slightly beyond this point. The ratio of increment of time to increment of bromate solution is plotted against the total volume of bromate solution already used, and the end-point of the titration is found from this curve. Each c.c. of 0·1 *N* bromate solution is equivalent to 0·004803 grm. of furfuraldehyde. The amount of the aldehyde present does not affect the accuracy of the method; hydroxymethylfurfuraldehyde and lævulinic acid, decomposition products of hexoses, do not interfere, but methylpentoses (*e.g.* rhamnose) and methylpentosans have an influence on the results obtained since they yield methylfurfuraldehyde which is titrated by the bromate solution.

W. P. S.

Oil from Kauri Copal. A. G. Hill and D. Nishida. (*J. Ind. Eng. Chem.*, 1923, **15**, 1276–1277.)—A dark yellow, viscous oil is obtained when Kauri copal is distilled at 330 C. under reduced pressure; the oil, known by the commercial name of "gum spirits," has the following characteristics:—Sp. gr. at 20° C., 0·9667; flash point, 85–86° C.; $[\alpha]_D^{20}$, +3·66°; n_D^{25}, 1·5128; acid value, 69·0; saponification value, 83·0; iodine value (Hanus), 114·0. After the oil has been washed with alkali solution, dried, and re-distilled under reduced pressure it has sp. gr., 0·9280; $[\alpha]_D^{20}$, +2·76°; n_D^{25}, 1·5102; iodine value (Hanus), 104. The oil, when exposed for six days in the form of a thin layer to the atmosphere, gives the colour reaction of rosin oil with the Liebermann-Storch test, and solidifies to a resinous film.

W. P. S.

Inorganic Analysis.

Estimation of Carbon Disulphide by Iodimetry. É. André. (*Bull. Soc. Chim.*, 1923, **33**, 1678–1681.)—The quantity of carbon disulphide corresponding with 1 c.c. of iodine solution is found to depend on the length of time during which the carbon disulphide and the alcoholic potassium hydroxide solution react. The reaction is evidently neither so simple nor so rapid as generally thought.

T. H. P.

Volumetric Micro-Estimation of Sodium. H. Müller. (*Helv. Chim. Acta*, 1923, **6**, 1152–1161.)—The serum (0·1 c.c.) is diluted with water to 1 c.c. in a small centrifugal tube, and treated with one c.c. of potassium pyroantimonate solution (2 grms. per litre), and, drop by drop, with 0·4 c.c. of 95 per cent. alcohol. The solution is centrifuged after 2 hours' standing, and the clear solution pipetted off. The precipitate is shaken three times with 2 c.c. of 30 per cent. alcohol, centrifuged each time, and the washings removed as before. The tube is heated at 80° to 100° C. on a water bath for the complete removal of alcohol. After cooling, the precipitate is treated with 1 c.c. of 2 per cent. potassium iodide solution per mgrm. of sodium assumed to be present, 1 c.c. of strong hydrochloric acid, and 2 c.c. of water; the solution is titrated, after ten minutes, with 0·01 N thiosulphate solution; the number of c.c. multiplied by 0·115 give mgrms. of sodium. Albumin need not be removed before the estimation, and the presence of phosphate, calcium, or magnesium does not affect the results. W. R. S.

Estimation of Sulphonitric and Sulphonitrous Acids. A. Graire. (*Comptes rend.*, 1923, 177, 821–823.)—The estimation of the nitrogen compounds in impure sulphuric acid cannot be effected by the permanganate method or by means of the nitrometer, since such procedure leads either to reduction phenomena or to the formation of complex salts with nitric oxide. On the other hand, Schlœsing's method, in which ferrous chloride is used, gives satisfactory results for the acids and oxides of nitrogen present. T. H. P.

Physical Methods, Apparatus, etc.

Identification of Minerals by Microscopic Examination of their Traces left on a Hard Body. P. Gaubert. (*Comptes rend.*, 1923, 177, 960–962.)—Information serving to characterise a mineral, partially or completely, is often obtainable by microscopic examination of the trace obtained by rubbing a fragment of the mineral on a ground glass slide or, for harder minerals, on a quartz plate cut perpendicularly to the axis and roughened with emery on one face. The transparency of the slide is restored by covering the trace with a liquid of about the same refractive index as the glass or quartz. Microscopic examination of the trace indicates the malleability, cleavage, friability, fracture, transparency, polychroism, bi-refraction, etc., of the mineral, and the refractive index may be estimated by using liquids with known indices. Further, in many cases, the degree of purity may be estimated from the appearance of the trace. This may also be treated with ordinary reagents and subjected to microchemical reactions. Thus, the presence of lead may be determined, from the form of lead nitrate crystals, in less than 0·00001 mgrm. of galena. Where doubtful results are obtained, comparison may be made with the traces left by known minerals.
T. H. P.

Burner for Producing Monochromatic Light. A. O. Jones. (*J. Soc. Chem. Ind.*, 1923, 459T.)—A one-inch Meker burner is arranged in such a way that its gas-supply has passed through a steel crucible heated by the burner and containing the desired volatile salt (sodium nitrite is convenient for a yellow flame).

The crucible has a screw cap with asbestos washer and is provided with steel side-tubes which support the crucible at a convenient height above the burner, and at the same time are connected directly with its gas-supply (sketch given). A short time after lighting the gas a strong monochromatic light is obtained.

R. F. I.

New Light Filter. L. W. McCay. (*J. Amer. Chem. Soc.*, 1923, **45**, 2958).—A filtered solution of ordinary chrome alum (310 grms. per litre) placed in glass bottles of square prismatic form (about 10 cm. high and 4·7 cm. at the lateral edge) forms an efficient light filter for observing the potassium flame. It absorbs completely not only the sodium light, but also that of lithium, strontium, calcium and barium. The solution is not only very sensitive, but will also keep indefinitely. It also shows the light of the rubidium and cæsium flames.

New Method of Gas Analysis. R. Geberth. (*J. Ind. Eng. Chem.*, 1923, **15**, 1277–1278.)—The method depends on the principle that mechanical energy impulses, such as sound waves, produced by a vibrating element, differ in character with the chemical composition of the gas through which they are transmitted. The apparatus used consists of a closed cylinder, adjustable as to length, through which the gases are passed, and having at one end a diaphragm coupled to a tuning fork and ammeter, the latter measuring the electric current operating the tuning fork. A graduated dial indicates the length of the gas chamber. The tuning fork is oscillated at a constant rate by an electro-magnet having a carbon transmitter as a variable resistance; as the column of gas is brought near to the resonance point, the ammeter, in series with the electro-magnet, indicates a very sharp rise in the current.

A change in the density of a gas sufficient to cause a change of 0·01 per cent. in the resonance length is readily indicated, and if the resonance length of one of the gases in a binary mixture is known, the composition of the mixture can be estimated. For instance, it is possible to indicate the presence of less than 0·1 per cent. of hydrogen in electrolytic oxygen, or of less than 1 per cent. of sulphur dioxide in air.

W. P. S.

Analysis of Mixed Salts by the Freezing Point Method. H. E. Batsford. (*J. Ind. Eng. Chem.*, 1923, **15**, 1272–1273.)—The method consists in fusing the salt mixture in an iron crucible and determining the freezing point by means of a thermocouple and recording instrument. A curve is plotted from the results obtained with mixtures of known composition, and this curve is used for reference in the analysis of mixtures of unknown composition. The triangular method of plotting ternary mixtures is illustrated. For example, a mixture of sodium chloride, calcium chloride and potassium chloride had a freezing point of 620° C. and contained 14·43 per cent. of calcium equivalent to 40·00 per cent. of calcium chloride; reference to the curve shows that the mixture also contained 46 per cent. of sodium chloride and 14 per cent. of potassium chloride.

W. P. S.

Method for Reproducing Graphs in Quantity. W. C. Greene and R. S. Hunt. (*J. Amer. Chem. Soc.*, **1923, 45,** 2961).—The graphs are plotted with waterproof drawing ink on the ordinary graph (or co-ordinate) paper. This is dipped into a saturated solution of a purified, colourless paraffin oil in chloroform, and, after about one minute, is withdrawn and allowed to drain. The excess of oil is then wiped off and the sheet left to dry in the air. The prepared paper is used as a negative for the reproduction of photographic copies on any of the bromide or blue print papers by direct printing.

Reviews.

ANALYTISCHE CHEMIE DER ALKALOIDE. By Professor K. H. BAUER. Pp. 425.
Berlin: Gebrüder Borntraeger. Price 30s.

This volume deals in a systematic way with methods for the recognition and the quantitative estimation of alkaloids, and it also contains a concise account of what is known of the chemistry of the alkaloids with which it deals. The author, perhaps wisely, makes no attempt to correlate the alkaloids in accordance with their chemical constitution, but classifies them by the source of origin, and the various groups of alkaloids are dealt with in haphazard succession.

The opening Sections deal with the many reagents which give precipitates with alkaloids, and the behaviour of the more important alkaloids with them. Colour reactions are also described, as well as other qualitative methods of identification.

In regard to methods of titration, the best indicators for use with the different alkaloids are given, and also some account of the varying neutral points obtained with different indicators. It is a pity that no reference is made to hydrogen ion concentration, or to the use of the newer indicators for this purpose.

To an English reviewer the book seems singularly incomplete, because it deals so very scantily with the important contributions which English chemists have made to this subject; indeed, the author does not appear to have had access to the Journal of the Chemical Society or to the ANALYST. We take haphazard two instances of the neglect of English work: no reference whatever is made to Dunstan's work on the aconitines, nor to that of Pyman on the emetine alkaloids. The old formula for emetine is employed notwithstanding the fact that it was corrected in 1914.

Professor K. H. Bauer has produced a book on a subject which is of importance to, and a book on which is needed by most analytical chemists. The volume is of the right size and is well printed. It is therefore disappointing to find that he has in a large measure failed to produce what is really wanted.

F. H. CARR.

THE MICRO-ORGANISMS OF THE SOIL. By Sir JOHN RUSSELL, F.R.S., and Others. Pp. viii.+188. London: Longmans, Green & Co. 1923. Price 7s. 6d. net.

Sir John Russell and his collaborators are to be congratulated on having brought together, in a small space and in an attractive style, the present knowledge of the interrelationship of the micro-organisms of the soil. The work is much enriched by an able historical review of the subject, and is illuminated by the contributions of the group of workers who have focussed their special knowledge on the soil problems.

In the words of the introduction, the purpose of the volume "is to give the broad outlines of our present knowledge of the relationships of the population of living organisms in the soil to one another and to the surface vegetation"; the text actually contains considerable detail which is augmented by a valuable bibliography. Of special importance is the work at Rothamstead, which has thrown so much light on the availability of the nitrogenous constituents of the soil. The influence of partial sterilisation on increasing the rate of oxidisation of the soil and the recognition of the important part played by the protozoa as a limiting factor in the bacterial numbers present in the soil, is of especial interest, and is dealt with in detail in the second chapter, on Soil Protozoa. Arising from this painstaking and laborious work great advances have been made in our knowledge of the distribution and activities of soil bacteria, ciliates, amœbæ and flagellates.

The "elective" methods of differentiating between the different groups of soil bacteria have made possible the study of many interesting groups in the "20,000,000 bacteria per grm. of soil which is now considered a fair average number."

The study of the organisms decomposing cellulose and their need for combined nitrogen has resulted in the development of a commercial process for making synthetic farmyard manure from straw, another contribution to practical agriculture from Rothamstead which may have a great economic future.

The complexity of the subject of nodule formation on leguminous plants, as disclosed by further work, still leaves room for improvement in our methods of legume cropping, conditions of which it is stated are being thoroughly worked out.

The Soil Algæ, Soil Fungi and the Invertebrate Fauna of the soil are ably discussed in other chapters of the book.

Much original work appears in the text, and it is obvious that full advantage is being taken of the unique facilities at Rothamstead for supplying historical, statistical and analytical data covering a long period of years.

In the final chapter, the Chemical Activities of the Soil Population and their Relation to the Growing Plant, are summarised in a very masterly manner.

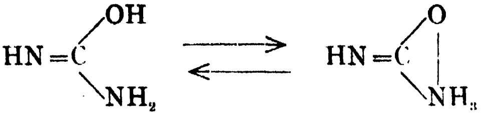
It is of special interest to note that each acre of dunged land loses, on an average, 41,000 calories per day, while each acre of unmanured land loses 2,700 calories per day. The human food produced on the dunged land yields only 7,000 calories per day.

The control of the soil population is still only in its infancy, but this useful contribution to our knowledge of the subject indicates the road and gives inspiration and a promise of useful development in future research work.

J. GOLDING.

THE CHEMISTRY OF UREA: THE THEORY OF ITS CONSTITUTION, AND OF THE ORIGIN, AND MODE OF ITS FORMATION IN LIVING ORGANISMS. By Prof. EMIL A. WERNER, M.A., Sc.D., F.I.C. (Monographs on Biochemistry). Pp. xii. +212. London: Longmans, Green & Co. 1923. 14s. net.

There is no one more competent to write on urea than Prof. Werner, and any book written by him on the subject must perforce be a valuable addition to the literature. It is, however, much to be regretted that Prof. Werner sets out mainly to prove his tautomeric formula



and to show that the old carbamide formula $CO(NH_2)_2$ is "quite untenable." Although it cannot be denied that he has made a good case for his formula, it must be left to the future to decide how far Prof. Werner has succeeded in convincing the chemical world at large as to the correctness of his views. There is, however, but little doubt that a more general treatment of the subject would have added greatly to the value of this monograph. As it stands, it is not the "Chemistry of Urea," but the case for the Werner formula.

M. NIERENSTEIN.

The Institute of Chemistry of Great Britain and Ireland.

PASS LIST.

JANUARY EXAMINATIONS, 1924.

THE following candidates have passed the examination for the Associateship:— *In General Chemistry:* D. S. Cohen, R. C. Doyle, H. C. Exell, H. H. Goldthorpe, B.Sc. (Lond.), A. T. S. Hare, F. P. Hornby, J. Johnston, B.Sc. (Lond.), A. N. D. Pullen, W. H. Radford, R. W. Watridge, B.Sc. (Lond.), and R. J. Wood.

(Nineteen candidates failed to satisfy the examiners.)

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THE ANALYST

10 APL

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ANNUAL GENERAL MEETING AND ORDINARY MEETING, HELD FEBRUARY 6, 1924.

THE Annual General Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, W.1. The President, Mr. P. A. Ellis Richards, F.I.C., was in the chair.

The Minutes of the previous General Meeting were read and confirmed.

Messrs. H. E. Burgess and T. McLachlan were appointed scrutators of the ballot papers for the election of Officers and Council for 1924.

The Hon. Treasurer presented the accounts of the Society for 1923, which were adopted, and votes of thanks were passed to the Hon. Treasurer and the Hon. Secretaries.

Messrs. Marreco, Houseman and Brandon, chartered accountants, were appointed auditors of the Society's accounts for 1924.

The President delivered his Annual Address. Dr. J. A. Voelcker proposed that a hearty vote of thanks be accorded to the President for his address and for his services in the chair, and that his permission be asked to print the address in THE ANALYST. This was seconded by Mr. E. M. Hawkins, and the motion was carried.

The Scrutators having reported the results of their examination of the ballot papers, the President announced that the Officers and Council for 1924 had been elected in accordance with the Council's nominations, as follows:—

President.—G. Rudd Thompson, F.I.C.

Past-Presidents serving on the Council (limited by the Society's Articles of Association to 8 in number).—Leonard Archbutt, F.I.C., A. Chaston Chapman, F.R.S., F.I.C., Bernard Dyer, D.Sc., F.I.C., Otto Hehner, F.I.C., P. A. Ellis Richards, F.I.C., Alfred Smetham, F.I.C., E. W. Voelcker, A.R.S.M., F.I.C. and J. Augustus Voelcker, M.A., B.Sc., Ph.D., F.I.C.

Vice-Presidents.—S. F. Burford, F.I.C., F. H. Carr, C.B.E., F.I.C., and Andrew More, A.R.C.Sc., F.I.C.

Hon. Treasurer.—Edward Hinks, M.B.E., B.Sc., F.I.C.

Hon. Secretary.—E. Richards Bolton, F.I.C.

Hon. Assistant Secretary.—R. G. Pelly, F.I.C.

Other Members of the Council.—F. W. F. Arnaud, F.I.C., H. Ballantyne, F.I.C., E. T. Brewis, F.I.C., R. T. Colgate, D.Sc., F.I.C., R. L. Collett, M.A., F.I.C., J. C. Drummond, D.Sc., F.I.C., S. Elliott, F.I.C., E. M. Hawkins, F.I.C., H. F. E. Hulton, F.I.C., A. E. Parkes, F.I.C., A. R. Tankard, F.I.C., and J. F. Tocher, D.Sc., F.I.C.

An Ordinary Meeting followed the Annual Meeting, the newly-elected President, Mr. G. Rudd Thompson, F.I.C., being in the chair.

Certificates were read for the first time in favour of:—Messrs. Philip Walter Alloway, Lorentz Oliver Brekke, B.Sc. (Leeds), A.I.C., Alex. Munro Cameron, B.Sc., F.I.C., Thomas William Drinkwater, L.R.C.P., L.R.C.S. (Edin.), F.I.C., John Ralph Furlong, Ph.D. (Wurzburg), A.I.C., Ernest Griffiths-Jones, M.Sc. (Manch.), A.I.C., Basil Gordon McLellan, F.I.C., and William Thomas Rigby, F.I.C.

Certificates were read for the second time in favour of:—Messrs. Hugh Browning Brown, A.I.C., Sidney Augustus de Lacy, A.I.C., A.M.I.Ch.E. Joseph Henry Lane, F.I.C., B.Sc. (Lond.), Leslie Herbert Lampitt, D.Sc. (Birm.), F.I.C., Reginald Francis Moon, B.Sc. (Bristol), Maximilian Nierenstein, D.Sc. (Geneva), Ph.D. (Berne), William Simpson Shaw, M.Sc., A.I.C., Robert Norman Wright, A.R.C.S., B.Sc. (Lond.), A.I.C., Misses Phyllis Honor Price, B.Sc. (Bristol), and Mabel Suzanne Lavinia Snelus, A.I.C.

The following were elected Members of the Society:—Messrs. Thomas Francis Doyle, George Hogan, F.I.C., Frank Knowles, Archibald Knox, A.I.C., Charles Roger Middleton, B.Sc. (Lond.), A.R.C.S., D.I.C., A.I.C., and Harold Richard Read, A.I.C., A.R.C.Sc. (Dublin).

The following paper was read:—"Osmium Tetroxide as a Reagent for the Estimation of Tannins and their Derivatives," by C. Ainsworth Mitchell, M.A., F.I.C.; and An Apparatus for Fat Extraction and Solvent Recovery was demonstrated by S. A. de Lacy, A.I.C.

STANDARDISATION OF ANALYTICAL METHODS.

The decision of the Council on this question is given in the President's Address (p. 123).—EDITOR.

Annual Address of the Retiring President.

SINCE I had the honour of addressing you in February last the Society has had a prosperous and successful year. Our members are now 465, as we have lost by death and by resignation 23 members, whilst 32 new ordinary members have been elected.

The names of the deceased members are:—George Embrey, Frederick James Lloyd, John Edward Stead, William Thompson, and John Matthew Wilkie.

George Embrey, who passed away on March 10th of last year at the age of eighty-three, was one of our oldest members. Born in Birmingham in 1840, he was educated at King Edward's Grammar School in that city, and as a student was trained at the Birmingham and Midland Institute and, afterwards, at the Royal College of Chemistry. He commenced his professional work as a consulting and analytical chemist in connection with the electro-plating and glass-making trades, and later moved to Gloucester, being appointed County Analyst in January, 1885, and City Analyst in September of the same year. He served as Member of Council of this Society for two periods, later becoming Vice-President, and in 1916 was elected President. His agreeable and genial nature endeared him to all his colleagues and his many friends deeply regret his loss.

Frederick James Lloyd was a member of this Society for many years, and also served on our Council. His early education was carried out at Bristol Grammar School, and in 1870 he entered Dr. Augustus Voelcker's laboratory, where he first studied agricultural chemistry. A little later he became assistant to Dr. Thomas Stevenson, and then senior assistant in the laboratory of the Royal Agricultural Society, finally starting practice on his own account in 1884. He will be long remembered for the special researches he carried out on the subject of Cheddar cheese-making and the chemistry of cider, whilst his loss will be felt not only by his colleagues in agricultural chemistry, but also by all members of the dairying industry, to whom his name was a household word.

William Thomson, who died very suddenly in his laboratory on October 4th, was for many years a member of this Society. Born in Glasgow in 1851, he commenced his professional career as assistant in the Royal Institution Laboratory at Manchester in 1869, becoming, four years later, a partner in the firm of Crace-Calvert and Thomson, and also Public Analyst for Stockport, an appointment he held until his death. He will be chiefly remembered for his work on the phosphorescent sulphides of calcium and barium, the detection of arsenic in beer, and for his efforts to diminish the soot pollution of the atmosphere of Manchester. His loss will be deeply felt by the many colleagues with whom he was closely associated.

John Edward Stead, another of our oldest members, who passed away at the age of seventy-two, was for long associated with analytical processes in relation to steel. In early days he studied under the late Mr. John Pattinson, and later joined him as partner. He devoted much time to microscopical metallography, and his great skill in this subject was widely recognised. In the past he was frequently present at our meetings, although, of late years, distance from London and his advancing age prevented his attendance. He was elected a Fellow of the Royal Society in 1903 and President of the Iron and Steel Institute in 1920.

John Matthew Wilkie commenced his career as a pharmaceutical chemist, and in 1900 entered the analytical laboratory of Messrs. Boots at Nottingham. For some years past he held the appointment there of deputy chief analyst, and, when the joint meeting of this Society and the Nottingham Section of the Society of Chemical Industry was held in that town last year, he took a very active part in the proceedings, and contributed a valuable paper on the estimation of minute amounts of arsenic. He passed away on November 29th after a severe operation.

During the year 1923 thirty papers have been read at our meetings, and the following have already been published in the pages of THE ANALYST:—

"The Colorimetric Estimation of Pyrogallol, Gallotannin and Gallic Acid." By C. Ainsworth Mitchell, M.A., F.I.C.
"The Estimation of Codeine." By H. E. Annett, D.Sc., F.I.C., and R. R. Sanghi.
"The Estimation of Narcotine and Papaverine in Opium." By H. E. Annett, D.Sc., F.I.C., and M. B. Bose, M.A.
"Note on the Sulphuric Acid Test for Fish Liver Oils." By Norman Evers, B.Sc., F.I.C., and H. J. Foster.
"Sliding Scales for the Convenient Titration of Strong Liquids by Dilution and the Use of Aliquot Parts." By C. H. D. Clark, B.Sc., D.I.C., A.I.C.
"The Estimation of Formaldehyde and Acetaldehyde." By E. W. Blair, B.Sc., D.I.C., A.I.C., and T. Sherlock Wheeler, B.Sc., A.R.C.Sc.I., A.I.C.
"Electrolytic Apparatus for the Estimation of Arsenic." By G. W. Monier-Williams, M.A., Ph.D., F.I.C.
"Some Observations with regard to the Unsaponifiable Matter and Sterols of Edible Fats." By D. W. Steuart, B.Sc.
"Notes on the Presence of Sulphur Dioxide in Cattle Foodstuffs after Fumigation." By H. Alan Peacock, B.Sc.
"The Examination of Firearms and Projectiles in Forensic Cases." By A. Lucas, O.B.E., F.I.C.
"Further Notes on the Estimation of Potassium by the Perchlorate and Cobaltinitrite Methods, and on the Removal of Sulphates." By R. Leitch Morris, F.I.C.
"The Action of Potassium Carbonate on Lead Glass." By H. Droop Richmond, F.I.C.
"Physiological Standardisation." By J. Stanley White, M.R.C.S., L.R.C.P.
"The Presence of Barium and Strontium in Natural Brines." By A. G. Francis, B.Sc., F.I.C.
"Titanium in Nile Silt." By E. Griffiths-Jones.
"An Investigation into the Chemistry of the Reinsch Test for Arsenic and Antimony, and its extension to Bismuth." By B. S. Evans, M.C., M.B.E., Ph.D., F.I.C.
"Determination of the Purity of Vanillin." By S. B. Phillips, A.I.C.
"The Quantitative Estimation of Hemp and Wood Fibres in Paper Pulp." By W. Dickson, F.I.C.

"The Estimation of Boric Acid in 'Liquid Eggs' and other Foodstuffs." By G. W. Monier-Williams, M.A., Ph.D., F.I.C.

"Notes on the Examination of Preserved Meats, etc." By Osman Jones, F.I.C.

"The Absorption of Atmospheric Gases by Water." By J. H. Coste, F.I.C.

"Tables for Sugar Analysis." By G. D. Elsdon, B.Sc., F.I.C.

"A Quantitative Study of the Limitations of the Reaction between Ammonia and Sodium Hypobromite." By D. R. Nanji and W. S. Shaw.

"Notes on a Method for Testing the Accuracy of Babcock Cream and Milk Test Bottles." By B. J. Smit, B.A.

"The Estimation of Lactose by the Polarimetric and the Gravimetric Methods." By A. L. Bacharach, B.A., A.I.C.

"Estimation of Fat, Lactose and Moisture in Dried Milks." By H. Jephcott, M.Sc., F.I.C.

"The Melting Point and Iodine Value of Refined Natural *d*-Camphor." By Maurice S. Salamon, B.Sc.

"Preservatives in Food: Hull Statistics for the ten years 1913 to 1922." By A. R. Tankard, F.I.C.

"The Sampling of Coal: The General Problem and some Experiments." By J. H. Coste, F.I.C., E. R. Andrews, F.I.C., and W. E. F. Powney, F.I.C.

"The Volumetric Estimation of Vanadium in Steel." By A. T. Etheridge, B.Sc., F.I.C., M.B.E.

"A New Test for Distinguishing Castor Oil from other Oils and Determining its Purity." By H. B. Stocks, F.I.C.

Our Journal, under the able editorship of Mr. C. A. Mitchell, continues as interesting and useful as ever. The last volume contains 634 pages of printed matter, compared with 544 pages for the preceding year, whilst, in addition to the original papers already referred to, we have published 20 notes, 521 abstracts, 57 reviews, and 32 legal and Parliamentary notes. Certain public analysts are sending us their reports regularly, and more of these would be welcomed. Government analysts overseas are co-operating in this regard, so that our Journal forms a connecting link between the work of those at home and those abroad.

Sixteen of the papers were concerned with the analysis of food and drugs, two with organic analysis, six with inorganic analysis, and three with apparatus and methods; there was one on bio-chemistry, one on forensic chemistry, and one on physiological methods of standardisation.

The Ministry of Health and other Government Departments continue to send us their Orders and Regulations, and these, or abstracts of them so far as they touch upon our work, are printed in THE ANALYST. A notable instance was the Report on the Loch Maree case of botulinus poisoning.

May I repeat the wish expressed last year that all papers on food analysis, toxicology, and bio-chemical work in connection with food products be contributed to our Society with a view to their publication in our Journal, the recognised medium for such communications.

Under the Analytical Investigation Scheme two papers have been read at our meetings during this session, one by Messrs. J. H. Coste, E. R. Andrews and W. E. F. Powney on "The Sampling of Coal," and the other by Mr. Harold Toms on "The Crystalline Bromides of Linseed Oil." Other investigations are proceeding, notably on fibre estimations required in paper analysis and on methods of estimating tannins. I hope that during the ensuing year some more of our members will find time to carry out further researches under this scheme, and, with this object in view, will put themselves in touch with our Honorary Secretaries.

Although the work was not undertaken in connection with the scheme, some interesting and useful tables for employment in sugar analysis have been drawn up by Mr. G. D. Elsdon and published in the September number.

A matter of considerable moment to both Public Analysts and consulting analysts generally has been the passing into law of the Milk and Dairies (Amendment) Act, 1922.

This Act definitely prohibits the addition of colouring matter or water or any dried or condensed milk, skimmed or separated milk, to ordinary milk intended for sale; and for the purposes of this section, except as regards the addition of skimmed or separated milk, milk includes skimmed and separated milk. This section is definite and helpful.

The Act further deals with milk sold under various designations, *i.e.* "Certified," "Grade A," and "Pasteurised," and licenses for the sale of milk are granted subject to certain terms and conditions prescribed by Orders made under this Act. In certain of these Orders, issued by the Ministry of Health, bacteriological standards for the various classes of graded milk have been prescribed. To secure compliance with these requirements, arrangements have been made for milk of these types to be systematically sampled by inspectors and examined bacteriologically. The Ministry expected to develop this work on a considerable scale, and hoped that public analysts would be prepared to undertake the necessary examinations. Since the latter are largely concerned with the control of the milk supply, the bacteriological examination would appear to many a natural extension of their work. But such examinations involve considerable time, labour, specialised knowledge and expense for materials and apparatus. For a suitable remuneration I have no doubt that many of us would have been prepared to undertake the work, but when, on enquiry, we found that the Ministry of Health had suggested that the fee should be five shillings per sample, the Joint Committee of this Society and the Institute of Chemistry were almost incredulous.

After careful consideration a joint deputation from the Institute and the Society was appointed to interview the Ministry on the matter. The results, however, were far from satisfactory, as the representatives of the latter stated very definitely that the scheme must be self-supporting, the only funds available being those derived from payments for licenses. The members of our deputation pointed out that the fee proposed would in no sense meet even the actual cost of the bacteriological examination, and, consequently, that no one of us would be prepared to undertake the work. We further made it clear that, in our opinion, an inferior rough-and-ready bacteriological examination was of no use at all, and was most unfair, not only to the general public, but also to the members of the dairy trade whose milk would be taken for testing purposes, as any lack of care or skill during the examination could only lead to an increase in the apparent bacterial content of the samples.

In conclusion we were informed that certain laboratories, the majority state- or rate-aided institutions, had undertaken to carry out the work at the suggested fee, but the Ministry would give very careful consideration to the views we had expressed.

In my address of last year I referred to Circular 325 issued by the Ministry of Health suggesting that it was undesirable that a prosecution should be based upon the results of an isolated test showing a deficiency of fat in the case of a particular milk supply. It is satisfactory to note that, following the joint letter of protest written by the Presidents of the Institute of Chemistry and this Society, the Ministry, recognising the unfortunate results produced by this suggestion, issued last May a further Circular withdrawing the former one.

The Ministry have issued Regulations as to the labelling and composition of condensed milk and of dried milk. Minimum percentages are fixed by the Regulations for fat and milk solids in full-cream condensed milk and for milk-solids in condensed skimmed milk. Similar minima are also fixed for full, three-quarter, half and quarter cream dried milk. It is also required in all cases that the instructions as to dilution or preparation given on a tin or package of condensed or dried milk shall be quantitatively accurate.

The Regulations in regard to condensed milk have already come into force, whilst those for dried milk are to operate as from May 1st, 1924.

Article 2, section 2, of the Regulations in respect of dried milk reads as follows:

"These Regulations apply to dried milk to which no other substance has been added, and to the dried milk contained in any powder or solid of which not less than 70 per cent. consists of dried milk."

Whether this section is intended to apply to sweetened dried milk only, or whether it is to cover such substances as infant's and invalid's foods is not clear.

Both of these Regulations have already appeared in our Journal, and so I have not attempted to give a *précis* of their requirements. The latter, valuable as they undoubtedly are in the public interest, necessitate considerably more work on the part of public analysts, but one regrets to see that the Ministry has made no suggestion for increased remuneration to these officers on this account.

In many counties and boroughs throughout the country it has been the custom to test the accuracy of the dispensing of medicines, the Food and Drugs Acts inspectors being instructed to submit definite prescriptions to pharmacists for this purpose. The contents of the medicine bottle are then divided into three parts, and one portion submitted to the public analyst in the ordinary way. Whether or no such samples come legitimately under the provisions of the Sale of Food and Drugs Acts is perhaps a moot point, but, assuming that they do, various troublesome facts arise that have to be dealt with by the analyst and taken into account by him when drafting his report.

In the first place many prescriptions usually given by medical men are not at all suitable for quantitative analysis. Again, the bottles in which the medicines are put up are notoriously very far from accurate, both as regards their actual capacity when full, and also as regards their sub-division into ounces, tablespoonfuls, and what not. It would appear to be a very general practice on the part of pharmacists, after weighing or measuring the required amounts of the various drugs, to make up to the total amount in the bottle rather than in a graduated measure. Consequently the final volume may be either greater or less than is supposed, depending on the actual capacity of the bottle. These and other points that I need not refer to here become of considerable importance when a question of prosecution arises.

In the spring of last year your Council was approached by the Council of the Pharmaceutical Society who proposed a joint conference on this subject. We welcomed this suggestion, and, as a result, a meeting took place at the rooms of the Institute of Chemistry between representatives of our Society, of the Pharmaceutical Society, and of the Retail Pharmacists Union. We had a very friendly, and, I think, useful meeting, the views of both sides being carefully considered and discussed. As an outcome, your Council suggested to the Ministry of Health that the latter should issue a Circular to Local Authorities recommending that the following procedure should be adopted in taking samples of prescriptions:

That the Analyst and the Medical Officer of Health should both be consulted as to the type of prescription it is desirable to use for the purpose of checking the accuracy of dispensing, and that, in the event of any substantial inaccuracies being disclosed by analysis, both these officers be consulted as to the desirability of instituting proceedings.

Further, that the inspector be instructed, prior to dividing the sample into three parts, to mark, in the presence of the vendor, the height that the contents reached in the bottle in which the medicine is originally supplied to him by the vendor.

Also that the bottle so marked be submitted to the analyst in order that he may determine the total quantity of medicine supplied.

The latter suggestion was made on account of the definite statement of the pharmaceutical representatives that the medical man, in writing out his prescription, intends that an aliquot portion of the total volume should be taken by the patient in each dose, rather than an accurate ounce, or half-ounce, etc., as the case may be. I am glad to say that the Ministry adopted the suggestions we made, and has issued to Local Authorities a Circular on the lines recommended.

With the letter already referred to, your Council sent another to the Ministry of Health pointing out that the analysis of dispensed medicines was never contemplated in the Act of Parliament, and that the fee customarily paid for analyses of samples taken under the Sale of Food and Drugs Acts cannot be considered a reasonable one in such cases. They urged the Ministry to use such means as they thought fit to ensure that the public analyst should receive adequate remuneration for the difficult work entailed by the analysis of such medicines. They further reminded the Ministry that, year by year, increased demands have been made upon the public analyst without any corresponding increase in his fee, and that to require work of this kind is putting an unjustifiable burden upon him.

I am sorry to say that the Ministry have not accepted this suggestion, and it remains for us to consider what further steps we should take in the matter.

This year I have to refer again to the entirely inadequate remuneration so frequently offered when a vacancy occurs in an official position under the Sale of Food and Drugs Acts. In the early part of last year the County and City of Gloucester advertised for a Public Analyst in the place of the late Mr. George Embrey. The number of samples proposed to be taken for analysis was not mentioned in the original notice, but enquiry elicited the fact that they were out of all proportion to the remuneration offered, and a protest was sent by the Joint Committee of the Institute and our Society to the Clerk of the Gloucestershire Council. In spite of this, the appointment was made on the terms proposed, but the successful candidate was not a member of our Society or of the Institute of Chemistry. The Ministry of Agriculture and Fisheries and the Ministry of Health have since informed the Gloucestershire County Council that they were not prepared to approve this appointment. A sequel to this is that the County Council is now exercising its legal right to send the work to the Public Analyst of another district, and it is understood that it intends to follow this course for such time as would afford the original candidate an opportunity of qualifying for the post.

Amongst other public analystships that became vacant during the past year were those for the City of London and the Borough of Islington. Your Council and that of the Institute of Chemistry regarded the terms offered in both of these cases as inadequate, and the joint committee again brought pressure to bear upon the authorities concerned with a view to improving them, but again the new appointments were made at the old rate of payment.

During the year our special branch of science has been brought somewhat more prominently than usual before the notice of the general public by the holding of various conferences and meetings in various parts of the country.

The Fourth International Chemical Conference took place at Cambridge under the presidency of Sir William Pope, and was a well attended and successful meeting.

In September the British Association met at Liverpool, and the brilliant address of the President, Sir Ernest Rutherford, on the electrical structure of matter has been received with world-wide interest, whilst many other important and valuable papers were contributed to the Chemistry Section.

Upon the invitation of the Liverpool and North-Western Section of the Institute of Chemistry a Conference of Fellows and Associates was also held there in October last. At this meeting several interesting discussions took place on matters of considerable moment to ourselves, in which many chemists, some of them members of our Society, took part. Amongst the subjects discussed were the questions of the formation of a national federation of men of science and the desirability or otherwise of a publicity campaign on the service to the community rendered by chemists.

In the same month a Chemists' Dinner, which was widely attended by the members of the various Societies, took place in London. This was in the nature of an innovation, as no dinner on exactly similar lines had been organised previously, and I am sure that all those who were present will agree that it was a most successful function, and one that should tend to increase the harmony and friendship already existing between those of us who are working in the many different branches of our profession.

During the year the Minister of Health has appointed a Committee to enquire into the use of preservatives and colouring matters in food and to report:

(1) Whether the use of such materials, or any of them, for the preservation and colouring of food is injurious to health, and if so in what quantities does their use become injurious;

(2) Whether it should be required that the presence of such materials and the quantities present in food offered or exposed for sale should be declared.

As originally constituted, this Committee included amongst its members as chemical representatives one of our past Presidents, Mr. Otto Hehner; one of our honorary members, Dr. F. Gowland Hopkins; and Mr. George Stubbs. Owing to the regretted departure of Mr. Hehner for South Africa, in November last, I was asked to fill the vacant seat on the Committee.

The matters referred to us cover a wide area, and yet in some respects, perhaps, the area is hardly wide enough, since the contamination of food with such substances as tin, lead, arsenic, etc., scarcely comes within our purview, although it is a matter of considerable importance to everyone.

Much evidence covering both the scientific and trade points of view has been and is still being heard, and it is to be hoped, for the sake of the public, and also especially for members of this Society, that when the Report of the Committee is issued, in due course, it will help to put the question of the use of preservatives and colouring matters in food on a more satisfactory basis than it is on at the present time.

With the state of the law now existing the procedure adopted by Local Authorities in regard to preservatives varies throughout the kingdom. In some districts no samples are taken with a view to the detection of preservatives, whilst in other places the vendors of foods so treated are prosecuted in almost all cases. This means, of course, that the public in places coming under the first category have no protection at all in this respect, whilst elsewhere they may be called on to pay the costs of actions rendered unsuccessful by the lack of standards and regulations.

Another point that requires very careful consideration is the importation into this country of food that, from necessity or not, as the case may be, comes in chemically preserved. Whether this treatment should be permitted, or what limitations should be placed upon it, needs a thoughtful and wise decision.

In this connection I may remind you that last month we held an extra meeting of this Society at the Institute of Chemistry, and devoted it to a discussion on preservatives in food. The debate was opened by Sir William Willcox and Dr.

Roche Lynch from the physiological, and by Mr. Hinks from the analytical standpoint. We had a very large audience of members and their friends, there was a good discussion, and the meeting proved a most interesting one.

The question of the advisability of standardisation of analytical methods has been under consideration by your Council for some time past, and the matter was referred finally to a sub-committee, who in due course made two recommendations to the Council, which the latter adopted. They are as follows:

> (1) The formation by the Society of a Committee to deal with the question of standard analytical methods in general is not considered practicable owing to the very large amount of specialised work which would be involved. The Committee regret their inability to advise the Society to undertake the responsibility of recommending standard methods.
>
> (2) The Committee recommend that chemists and analysts interested in particular industries be invited to confer among themselves upon cases where divergent results have occurred owing to the use of different methods of analysis, and to submit reports to appropriate committees of the Society; these reports, after consideration, to be published in THE ANALYST; the Society, however, taking no responsibility for recommending methods of analysis, but merely acting as a "clearing house."

Following this, a paper on "The Plea for Standardisation" was contributed by Mr. M. S. Salamon to the December meeting of the Society, and an interesting discussion ensued.

I believe I have referred to all the chief events that have occurred in connection with, or are of interest to, the Society during the past year, and must now bring this address to an end. It has, I fear, of necessity, been a little disjointed, as so many varied topics have had to be touched upon.

To-day marks the conclusion of my eighteen years official connection with the Society, first as Honorary Secretary, and for the last two years as your President. It is a somewhat long period, upon which I can look back with great pleasure on account of the uniform kindness and consideration extended to me by all members of the Society, and more particularly by the Officers and Council, with whom my duties have brought me most in contact. As President, my work has been comparatively light on account of the able assistance of the Officers, Messrs. Mitchell, Hinks, Bolton and Pelly, to all of whom I tender my most hearty thanks.

Before I resume my seat I desire to welcome most cordially as my successor my old friend, Mr. G. Rudd Thompson. In Mr. Thompson you will have as President one who has the interests of the Society closely at heart, who has been long connected with it, and who will, I am sure, occupy the chair with credit and distinction to the Society and himself.

The Estimation of Lead in Potable Waters, and in Urine.

BY JOHN C. THRESH, M.D., D.SC., F.I.C.

(Read at the Meeting, November 7, 1923.)

IN a previous paper, published in the Journal of the Society for July, 1921 (ANALYST, 1921, **45**, 270), a method was described for estimating small amounts of lead in water, and it was pointed out that variations in the acidity caused a variation in the colour produced by colloidal lead sulphide. Further use of the method confirms the opinion expressed of its reliability, but it seems preferable to make the gelatin acid solution as follows:

Acetic acid B.P., 30 c.c.; distilled water, 70 c.c.; and gold leaf gelatin, 0·1 grm. Dissolve the gelatin in the dilute acid at a temperature below the boiling point. The amount used in each experiment is 1 c.c. to 50 c.c. of the water to be examined.

Unfortunately most moorland waters are coloured, some markedly so, and this colour interferes with the estimation of the lead. At first the difficulty seemed to be surmounted by using the same water, or the same dilution with distilled water, in making the standard solutions, but, under certain circumstances, it is not always as satisfactory as could be desired, especially when the amount of lead to be estimated is very small. Also, it soon became obvious that different waters gave different depths of colour when containing equal quantities of lead, and further investigations shewed that different distilled waters gave variable results. The following table exhibits the extent of this variation and the extent of the error which would result therefrom: All the water used contained 0·2 mgrm. of lead in 100 c.c.

	Colour.	Lead sulphide indicated Mgrm.
(1) The purest distilled water, glass condensed ..	1·0	0·2
(2) The purest distilled water condensed in tinned copper	0·3 to 0·9	0·06 to 0·18
(3) Commercial distilled waters	0·6 to 1·0	0·12 to 0·2
(4) Moorland water, Durham	0·7	0·14
(5) ,, ,, Central Wales	0·8	0·16
(6) ,, ,, Cumberland	1·0	0·2
(7) Infusion of heather rootlets (very dilute) ..	1·0	0·2
(8) ,, of bog moss (very dilute)	0·85	0·17
(9) ,, bilberry rootlets (very dilute)	0·8	0·16

It will be observed that moorland waters do not vary nearly so much as distilled waters, and it was a long time before the cause of the variation was discovered; in fact, it was long before it was discovered that the nature of the condensing apparatus was chiefly responsible. This delay was due to the extraordinarily small amount of impurity which can produce a marked effect. On connecting a boiler with two condensers, one of glass and the other of tinned copper, it was found that the water collected from the former was uniform and satisfactory, whilst that collected from the latter varied considerably, and the more slowly the water was distilled the worse the result. Finally it was found that copper was the interfering substance. With hydrogen sulphide copper gives a much paler colour than the same amount of lead.

Colour given by
H_2S with 0·2 mgrm. of lead in 100,000 .. .. Colour 1·0
,, ,, 0·2 mgrm. copper in 100,000 .. .. ,, 0·3
,, ,, 0·2 mgrm. copper+0·2 mgrm. lead+0·2 mgrm. copper ,, 0·5

Hence, instead of the copper increasing the depth of tint, it reduces it fifty per cent.

The effect of still smaller quantities is shewn in the following table, where different quantities of copper were added to water containing 0·2 mgrm. of lead in 100,000.

Copper added per 100 c.c.	Colour corresponded to distilled water + lead.	Colour corresponded to Cumberland water + lead.
Mgrm.	Mgrm.	Mgrm.
0·1	0·1	—
0·05	0·06	—
0·025	0·06	—
0·01	0·9	—
0·005	0·12	0·14
0·0025	0·14	0·16
0·0015	0·19	—
0·001	—	0·1
0·000	0·2	0·2

The presence of one part of copper in 100 millions has a distinct effect.

Copper was found in the distilled waters giving too little colour with lead sulphide, but it has only been found in one of the samples of moorland water examined. This was from South Wales, and contained 1 part in 100 millions.

This amount has a decided effect, but if metallic tin is immersed in a water containing a trace of copper the copper seems to be removed, as the water then gives a normal reaction with lead sulphide. Silver, on the other hand, acts very much like copper. A silver spoon left all night in a pure distilled water has a marked effect in reducing the colour of colloidal lead sulphide. The above results, however, did not explain the variations with moorland waters free from copper. Further investigation shewed that the interfering substance was not of organic nature, but simply iron in solution. This is not so powerful as copper, but it explains all discrepancies, since every coloured moorland water examined contained easily detectable traces of this metal. The effect of small quantities is shewn in the results obtained with various dilutions in distilled water containing 0·2 mgrm. of lead in 100,000.

Iron present. Mgrm.	Colour.		Lead.
0·1	0·3	=	0·06
0·05	0·3	=	0·06
0·02	0·35	=	0·07
0·01	0·7	=	0·14
0·005	1·0	=	0·2

Hence, in the case of a water which, undiluted, gives any colour with the potassium thiocyanate test for iron, the iron present will appreciably effect the estimation of the lead.

It is obvious, therefore, that, in estimating the amount of lead in any potable water, a sample of the same water free from lead should be obtained for making the standards for comparison.

The importance of the standard and water examined having the same degree of acidity and acid of identical character may again be emphasised, especially as it has a marked bearing upon the estimation of lead in urine. The subjoined table will impress this fact upon the mind.

Lead in each solution 2 mgrms. in 100 c.c.

Acid added to 100 c.c.	Colour.
2 c.c. Gel. Acetic acid	1·0
4 c.c. ,, ,, ,, ...	0·85
2 c.c. 0·1 *N* hydrochloric acid	0·75
2 c.c. *N* hydrochloric acid	0·0

With 2 c.c. of 0·1 *N* hydrochloric acid the colour is different from that produced by the gelatin acetic acid, and in a very few minutes the water becomes opalescent, and accurate comparison is impossible. The colouring matter of peaty waters always contains iron, and apparently the colour is always reduced by acidification, and increased (to a less extent) by addition of an alkali. A number of experiments have been made comparing this colour with that given by dilute infusions of *Sphagnum* and of rootlets of heather and bilberry. The colour apparently comes from the two latter (often chief constituents of peat), but some of the free acid is derived from the *Sphagnum*, and these acids are probably quinic acid and some product of the oxidation of tannin. This question, however, is left over for further investigation.

From what has been already said it is obvious that the detection of very minute quantities of lead in coloured liquids must be a matter of some difficulty. In connection with suspected cases of lead poisoning in a town using a moorland water, samples of urine from the patients under observation were submitted to examination. It was found that the addition of acid and hydrogen sulphide to the urine never produced any obvious change in colour, whereas a change could be detected if the urine contained 1 part of lead in 2 millions; the amount present, if any, must have been less than this. No satisfactory test could be found in the text books, and, accordingly, a series of experiments was made to devise one. At first, advantage was taken of the fact that water or urine containing lead gives this metal up very rapidly, even in the presence of dilute acetic acid, to specially prepared charcoal, but it was found that the charcoal contained a trace of iron and retained the lead somewhat obstinately. Finally its use was abandoned, and the following method was devised, which gives excellent results in skilled hands:

Heat a measured quantity of the urine (not less than 100 c.c.) nearly to boiling point, add *N* sodium carbonate solution until the liquid becomes turbid, and the precipitate flocculates on gentle agitation (1 c.c. per 100 c.c. of urine suffices in most cases). Then allow the whole to stand until the supernatant liquid is clear. This can be poured off or passed through a small filter paper, upon which the whole precipitate is collected, washed with distilled water made faintly alkaline with sodium carbonate, and finally dissolved in about 2 c.c. of B.P. acetic acid, plus 10 c.c. of hot water. The liquid and washings need not exceed 25 c.c. Then add 0·5 c.c. of gelatin acetic acid and hydrogen sulphide. The colour then produced must be estimated from a solution of corresponding strength of acid.

No one should attempt this estimation until he can get absolutely negative results with lead-free urine treated in the above manner, but, if this cannot be done, the amount found in the check examination may be deducted from that found in the suspected urine. After assuring myself that the chemicals and glass ware used contained no lead, positive results were still obtained. Finally the filter paper was suspected and found to contain lead. Filter paper which has been thoroughly exhausted with acid must therefore be used.

I have had submitted to me the analyses of 3 kinds of filter paper usually employed for qualitative work.

	Ash. Per Cent.	Pb. Per Cent.	Cu.
1. English make	0·17	0·0078	None.
2. A continental make	0·41	0·0106	Very slight trace.
3. Another continental make ..	0·53	0·0095	ditto.

The following results were obtained with urine to which lead had been added. The first series of analyses was made by myself, the second by a friend, who could not get a negative result with another urine.

Series 1		Series 2.	
Lead added. per 100 c.c. Mgrm.	Lead found. Mgrm.	Lead added. Mgrm.	Lead found. Mgrm.
0·1	0·105	0·00 (check sample)	0·03
0·075	0·08	—	—
0·05	0·035	0·04	0·07
0·05	0·058	Diff.	0·04
0·04	0·043	0·00 (check sample)	0·02
0·02	0·03	0·03	0·05
0·02	0·021	Diff.	0·03
0·01	A trace.	—	—

When urine, not distinctly acid, containing a trace of lead, is passed through filter paper, most, if not all, of the metal is retained by the fibres of the filter, and can be dissolved out with acid, but, as a method of detecting and estimating lead, the results obtained were not sufficiently concordant to render the process available.

LEAD IN BEER AND CIDER.—In attempting to estimate small quantities of lead in beer and cider by the process used for urine, it was found that the precipitate produced carried down so much colouring matter that the process was not applicable. By using precipitated chalk, instead of sodium carbonate, the precipitate is very pale in colour, and the acetic and solution, though not colourless, can be used for nesslerising, a dilute solution of burnt sugar being used for the standard. In this way a beer to which 1 part of lead per million had been added gave a most distinct reaction with hydrogen sulphide, and the estimated amount of lead was 0·08. With some slight modification, it is obvious that this method may be useful.

DISCUSSION.

Mr. W. T. BURGESS said that the author's paper made us wonder whether our colorimetric estimations were correct. He was surprised to find that if one had a little copper in the presence of lead the colour was only the same as when lead alone was present. Anyone with experience of water knew that peaty water changed its colour when its reaction was altered by the addition of acid or alkali. He called attention to the organic matter in the reagent used by the author; the gelatin put into the test was 200 parts per 100,000. He doubted whether these tests would come out in this way when hydrogen sulphide alone was used.

Dr. Thresh had asked him whether he had suspected lead in the filter paper; he had not done so, but he had found it useless to filter water containing traces of lead through filter paper, owing to the adsorption of the lead by the filter paper.

Mr. RAYMOND ROSS said that he would like to know why it was necessary to estimate the lead as sulphide and not chromate; with the latter, he considered, it would be easier to judge the opalescence. If water was being passed through a mechanical filter it was always best to filter it on the acid side, because, if one allowed it to become at all alkaline, when passing through the filter, it would absorb colour from the filter; if it was acid, one could add lime water, after filtration, without altering the colour.

Mr. E. M. HAWKINS, referring to the sulphide test, said that the reaction was much affected by a very minute quantity of acid.

Mr. E. J. GUILD, referring to the peaty water of Edinburgh, said that he agreed with the author as to the difficulty of estimating lead in the presence of iron; as regards filter papers, they had actually estimated the amount of lead by ashing and precipitation of the lead as sulphate. He emphasised the importance of making blanks, especially in the case of a delicate test.

The Colorimetric Estimation of Lead in Cream of Tartar.

By R. L. ANDREW.

(*Read at the Meeting, December* 5, 1923.)

In connection with the administration of the Sale of Food and Drugs Act all importations of cream of tartar during 1921–1922 were examined for lead.

This resulted in the carrying out of a large number of estimations, and involved a critical examination of methods.

The B.P. method, with the use of primary and auxiliary solutions containing seven and two grms. of tartrate respectively, is cumbersome, and has the further objection, that, in the case of samples carrying large amounts of lead, the colorations produced are so intense that accurate comparisons are difficult.

A more satisfactory method is to take two grms. of the cream of tartar, add 1 c.c. of 5 per cent. potassium cyanide solution, 1 c.c. of ammonia, and 40 c.c. of water, and dissolve by warming and shaking. The solution is cooled and passed through a dry filter.

Standards are prepared in a similar manner from two grms. of lead-free cream of tartar, with the addition of requisite amounts of a lead nitrate solution containing 0·01 per cent. of lead. Any tint in the solution is then matched by the addition of very dilute caramel to the standards.

A few drops of ten per cent. sodium sulphide solution are added, the solutions made up to the mark, and the amount of lead estimated by comparison with the standards.

The results so obtained agree with those of the B.P. method, using 7 and 2 grms. of cream of tartar, shewing that the different concentrations of tartrate do not affect the depth of colour of the lead sulphide.

If, however, an aqueous solution containing lead is used for the standard, the colour is only about half the depth of that produced by an equal amount of lead in a tartrate solution, and a serious error is introduced.

The effect of iron in both the ferric and ferrous states was investigated.

R. R. Tatlock and R. T. Thomson (ANALYST, 1908, 33, 176) say that the iron should not exceed 0·25 mgrm. in 50 c.c. of the solution under examination; otherwise the results may be on the high side.

It was found that amounts of iron not in excess of 0·4 mgrm. have no effect.

Contrary to Wilkie's findings (*J. Soc. Chem. Ind.*, 1909, 28, 636), it makes little, if any, difference whether the iron is in the ferric or ferrous state.

An amazing error was found in the method given for the detection of lead in cream of tartar, in Allen's *Commercial Organic Analysis*, 4th edition, Vol. I., p. 549, where it is recommended that "if the solution is coloured it must be treated with purified animal charcoal and filtered." Such treatment completely removes any lead that may be present. Experiments shewed that 0·5 grm. of animal charcoal will, under the conditions of the estimation, remove 1 mgrm. of lead from solution.

Of 389 samples of cream of tartar examined during 1921 and 1922, only 26 contained more than one-seventh of a grain of lead to the pound.

Many of the samples were of a very high degree of purity, some failing to shew a trace of lead in ten grms., and in no case was there sufficient iron present to affect the estimation.

No originality is claimed for these observations, but experience has shewn that sometimes sufficient attention is not paid to possible errors, particularly to that produced by the use of an aqueous solution of lead as a standard.

Dominion Laboratory,
Wellington, New Zealand.

Notes on the Estimation of Chromium.

By HUBERT T. S. BRITTON, M.Sc., F.I.C.

(Read at the Meeting, December 5, 1923.)

I. Gravimetric Estimation.—The gravimetric estimation of chromium by precipitation as the hydroxide and subsequent ignition to the oxide does not give quantitative results. They are always too high, usually about one or two per cent. Prolonged heating of the oxide at a high temperature appears to be one of the conditions necessary to eliminate this error. The reason for these high results is to be found in the fact that the ignited oxide invariably contains appreciable amounts of chromic anhydride, as may be seen when the ash is extracted with water and the solution tested for chromate by the ordinary reagents.

This observation is by no means new. There appears, however, to have been some controversy extending over the last sixty years concerning the magnitude of the error involved. Treadwell, on page 102 of the 1919 English edition of his *Quantitative Analysis* (trans. by Hall), states that "the results obtained are always a few tenths of a per cent. too high on account of the formation of small amounts of alkali chromate, even though the entire operation takes place in platinum vessels. The alkali comes from the reagents." When every precaution is taken to use only the purest reagents and salts, the ignited oxide is still found to contain some extractable chromate. This was also the experience of Rothaug. (*Zeitsch. anorg. Chem.*, 1914, **84**, 165).

Souchay (*Zeitsch. anal. Chem.*, 1865, **4**, 66) was the first to observe that gravimetric analysis gave high values, but, as he happened to obtain figures 2·62 per cent. too high when the precipitation was carried out in glass vessels, as compared with 1 per cent. in platinum vessels, he was led to conclude that the arger increase was due to some extraction from the glass having taken place. Similar views were held by Wilm (*Ber.*, 1879, **12**, 2172 and 2223), whose results showed errors up to 6 per cent. On the other hand, Treadwell (*Ber.*, 1882, **15**, 1392) obtained accurate results, no matter in what vessels the estimations were made, although he mentions that the ash always contained some chromic anhydride. Later, Stock and Massacui (*Ber.*, 1901, **33**, 467) apparently obtained accurate results when the precipitation was carried out by means of a mixture of potassium iodide and iodate. In 1914 Rothaug (*loc. cit.*) published a very exhaustive paper on the estimation of chromium as oxide. He investigated the methods in which the chromium is precipitated with each of the following reagents: Ammonium hydroxide, ammonium sulphide, and a mixture of potassium iodide and iodate. With ammonia his results show an average error of 0·75 per cent.; with ammonium sulphide, 1·6 per cent.; and with the iodide-iodate method 1·02 per cent. After heating the oxide in an electric furnace for two hours the error was much less, averaging 0·17 per cent. The only procedure which gave him accurate results was ignition of the oxide in a stream of hydrogen. Analyses of aqueous extracts of the ash showed, on ignition, some chromium chromate—$Cr_2(CrO_4)_3$—was formed, and was more or less decomposed by heating at a higher temperature, and completely reduced to chromium oxide when heated in an atmosphere of hydrogen. Hence, it will be inferred that as the ash on ignition tends to form coherent particles, thereby enveloping some chromate, the total decomposition of the higher oxide will be an extremely slow process.

As these observations happened to be in accord with the author's experience, the errors being much greater than would be gathered from the remarks of Treadwell, it was resolved to direct attention to volumetric methods.

II. VOLUMETRIC ESTIMATION.—Several methods of oxidation of chromic salts to chromic acid have been put forward, but they involve the use of such reagents as hydrogen peroxide and persulphates. Thus W. J. Sell (*Chem. News*, 1886, **54**, 299) precipitated the chromium with potassium hydroxide and then added more alkali in amount just sufficient to redissolve the precipitate. To this solution an excess of hydrogen peroxide was added, and the liquid then boiled to complete the oxidation, and also to decompose the excess peroxide. Afterwards the chromic acid was estimated iodimetrically. This method of oxidation was examined by Bourion and Sénéchal (*Comptes rend.*, 1913, **157**, 1528), who found that a considerable excess of hydrogen peroxide is necessary to ensure complete oxidation, and that the excess of alkali is without much effect on the decomposition of the excess peroxide. They estimated the chromic acid thus formed by allowing it to oxidise ferrous ammonium sulphate and titrating the unoxidised iron with potassium permanganate solution.

Up to the present, no one appears to have used sodium peroxide, as such, in the volumetric estimation of chromium, although less common and more troublesome substances have been used.

The following is a description of some work done in which the conditions for the accurate volumetric determination of chromium were established. Solutions of sodium peroxide of about 1 per cent. concentration are decomposed quantitatively after boiling for ten minutes; the amount of iodine which is liberated from potassium iodide after the solution has been rendered strongly acid in the cold is beyond the limits of titration. Again, chromium salts are readily oxidised to chromates by means of sodium peroxide in alkaline solution, but in acid solutions the addition of peroxide brings about the formation of perchromic acid and the subsequent reduction to the original condition. Thus, all that is necessary is to oxidise in alkaline solution, decompose the excess of peroxide by boiling, cool and then acidify with either moderately concentrated hydrochloric or sulphuric acid, add potassium iodide and, after dilution, titrate the liberated iodine with sodium thiosulphate.

The following procedure was found to yield readily accurate results: The acid present in a solution of the chromium salt is roughly neutralised, and an excess of sodium peroxide (about 1 grm. is generally sufficient) is added. The solution is diluted to about 100 c.c. and boiled for ten minutes, in which time the chromium is completely converted into chromate and the excess of peroxide decomposed. A precipitate sometimes appears during boiling, but re-dissolves as the oxidation proceeds. After cooling, the solution is acidified, and the chromic acid estimated iodimetrically. If the solution has been sufficiently cooled, it does not matter whether hydrochloric acid or sulphuric acid is used; but if the solution is at all warm there is a distinct tendency to obtain low results with hydrochloric acid, on account of the oxidation of the hydrochloric acid by the chromic acid.

RESULTS.—The following are some results which were obtained by this method, the chromium having been estimated in varying quantities of pure chrome alum:

Weight of chrome alum taken. Grm.	Equivalent to Cr_2O_3 taken. Grm.	N/20 $Na_2S_2O_3$ required. c.c.	Cr_2O_3 found. Grm.
0·1108	0·0153	12·10	0·0153
0·2246	0·0342	27·10	0·0343
0·2288	0·0348	27·60	0·0350
0·2230	0·0339	26·85	0·0340
0·4000	0·0609	47·93	0·0608
0·6993	0·1064	83·85	0·1062

The following typical results (as Cr_2O_3) were obtained gravimetrically by precipitation with ammonium hydroxide and by the more accurate volumetric method: Gravimetric, 0·1096; volumetric, 0·1074 grm. Here the gravimetric result is 2·04 per cent. too high.

IMPERIAL COLLEGE OF SCIENCE AND TECHNOLOGY,
SOUTH KENSINGTON.

NOTE.—Since writing the above, my attention has been directed to a paper by J. Clark (*J. Chem. Soc.*, 1893, 63, 1083) on the use of sodium peroxide as an analytical reagent. In the estimation of chromium in chrome steel, he obtained satisfactory results only when the oxidation was carried out by the usual fusion process. He stated, however, that the oxidation may be effected in solution by adding a freshly prepared solution of sodium peroxide to the chromium solution until it had become alkaline, and then boiling. He stated that this method yielded low results. This was evidently due to the fact that he had also much iron present in the solution, thereby causing some chromic acid to be occluded by the ferric hydroxide which was precipitated. He did not test the accuracy of the method when there were no disturbing influences present in the solution.

THE APPLICATION OF THE IODIMETRIC METHOD TO THE ANALYSIS OF SUGAR PRODUCTS.

BY C. L. HINTON, F.I.C., AND T. MACARA, F.I.C.

ERRATA.—Delete "C" after the polarisation degrees in the following places in the January issue:—P. 6, l. 8 and 9; p. 7, l. 29; p. 12, l. 24, 28, 30, 31.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

ESTIMATION OF BORIC ACID IN MILK, ETC.

SPECIAL experiments on Thomson's method (ANALYST, 1896, **21**, 64) proved that: (1) In the presence of excess of alkali boric acid is not volatilised at a red heat. (2) It is better to treat the ash with water and hydrochloric acid successively. (3) If the solution is not dilute, alkali will precipitate calcium borate. (4) The amount of calcium chloride recommended by Thomson (=7 c.c. of a *N* solution for 70 c.c. of milk) is too much, and the addition of lime water increases the precipitation of calcium borate. (5) The alternate addition of calcium chloride and sodium hydroxide in the presence of phenolphthalein, as recommended by Shrewsbury (ANALYST, 1907, **32**, 12) is a satisfactory indication of the correct amount of calcium chloride to be added in the absence of carbon dioxide. If carbon dioxide is not removed the end reaction is indefinite, and phosphate remains in solution. (6) The acid solution can be boiled for 5 minutes in an open flask without loss of boric acid.

The following is the method recommended:—Evaporate 70 c.c. of milk in a platinum dish on a water bath with 7 c.c. of 3 *N* sodium hydroxide solution for about four hours. Apply a flame to the side of the dish, taking care that the contents do not froth over. Complete the ignition at a high temperature until the contents are white, or nearly so. Add 35 c.c. of water and filter after standing. Add 5 c.c. of 3 *N* hydrochloric acid to the dish and 15 c.c. of water. Filter through the same filter, and wash dish and filter with 4 c.c. of 3 *N* hydrochloric acid mixed with 16 c.c. of water. Boil for 5 minutes, cool, and add 6 c.c. 0·5 *N* calcium chloride solution, phenolphthalein and *N* sodium hydroxide solution to make the liquid a permanent faint pink. Add another c.c. of calcium chloride solution and more sodium hydroxide if the colour is destroyed. Repeat this addition, if necessary. Dilute to 100 c.c. and filter through a dry filter. To 80 c.c. of filtrate add methyl-orange and 0·1 *N* hydrochloric acid to slight excess. Boil for 5 minutes, cool, neutralise to methyl-orange and titrate with 0·1 *N* sodium hydroxide solution and 30 c.c. of glycerin. Subtract the acidity of 30 c.c. of glycerin.

W/v boric acid =c.c. 0·1 *N* sodium hydroxide solution × 0·011.
Grains per gallon =c.c. 0·1 *N* ,, ,, ,, × 7·75.

If more than 5 c.c. of sodium hydroxide solution are used, calcium borate may be precipitated, and the precipitate should be re-dissolved and re-precipitated as before.

In the test analysis 5 c.c. of 0·1 *N* boric acid were added to 70 c.c. of milk, and 4·95 c.c. of 0·1 *N* sodium hydroxide solution were required, but when 10 c.c. of boric acid were used 9·7 c.c. were found, indicating that with this quantity re-precipitation is necessary. Genuine milk treated as above requires no 0·1 *N* sodium hydroxide solution.

The same method may be applied to preserved cream and cake, 10 grms. being taken, or more if the proportion of boric acid is small. With liquid eggs it is convenient to heat gently about 2 grms. with 50 c.c. of 1·5 *N* alcoholic sodium hydroxide in a covered dish for 2 hours, then to evaporate to dryness and ignite as before. Sausages may be treated similarly, about 10 grms. being used.

J. F. LIVERSEEGE.
H. H. BAGNALL.

44, BROAD STREET, BIRMINGHAM.

BORIC ACID IN CAKE.

IN reference to the Circular, No. 381, by the Ministry of Health (20–3–23), "Boric Acid in Cake," I would like to draw the attention of those of our members who are interested in the question to a somewhat startling experience which we have had recently arising out of the Circular in question, for, as is known, the Bakery and Allied Traders' Association gave a definite undertaking as to the boric acid content of imported whole egg and/or yolk; hence, on examining certain cakes, the boric acid found rather pointed to a breach of the above undertaking.

The cakes in question were various varieties of so-called fruit cakes, but, on picking out the currants, sultanas, raisins, dried cherries, etc., and separately testing them, we were much perturbed at the boric acid content of these dried fruits.

Further purchases of these articles, *per se*, in the ordinary shops, have shewn boric acid contents which would rather cause me to believe that we are dealing with something more than a natural constituent. This is purely a preliminary note to a detailed paper upon the subject, which is well in hand, and which we hope to present in due course to the Society, as it certainly opens up a rather wide field in (possibly) a hitherto unexplored region.

G. RUDD THOMPSON.

NEWPORT, MON.

THE RATE OF LOSS OF STRENGTH OF SPIRITS KEPT EXPOSED TO THE AIR.

In many cases when proceedings are taken for selling diluted spirits the defendants plead that the loss of alcohol is due to evaporation owing to the spirit being left exposed to the air.

In a recent case where this excuse was pleaded, the spirit had been kept in a small china barrel, the bung hole being covered with a piece of cardboard. The sample was found to be 37·5° under proof at 15·5° C. With a view to testing how rapid such loss of alcohol might be, 250 c.c. of whisky, of exactly 34·1° U.P. strength, were kept in a flask, with the mouth loosely covered with a cardboard cover, at room temperature, which did not exceed 15·5° C. throughout. The following results were obtained:

Length of time Days.	Strength of spirit at 15·5° C.
0	34·1° U.P.
1	34·5° ,,
2	34·9° ,,
3	34·9° ,,
4	35·1° ,,
21	37·1° ,,

In 21 days the spirit had fallen in strength 3°, an average of about 0·15° per day. The loss, no doubt, would vary with temperature and humidity of the atmosphere, but the above figures give some indication of what may be expected under normal conditions of temperature.

Harold Lowe.

Laboratory, Assay Office, Chester.

DEVICE FOR STOPPING A LEAK IN A WATER JACKET.

With the Haldane apparatus for air analysis, trouble is sometimes experienced on account of the water jacket developing a leak. The jacket consists of a glass cylinder closed at the bottom with a cork. Even a very marked leak can be immediately stopped by pouring in mercury to a depth of about ⅜"; this can be done without obscuring the graduations of the air burette. Presumably this method would be equally effective with any similar water jacket.

Robert C. Frederick.

Royal Naval Medical School,
Royal Naval College, Greenwich, S.E.10.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

LEAD STANDARDS FOR CREAM OF TARTAR.

On December 14 the Birkenhead and District Co-operative Society were summoned for selling cream of tartar not of the nature, substance and quality demanded, since it contained 44 parts of lead per million.

The Inspector admitted, in cross-examination, that he had asked for and received cream of tartar.

Mr. H. E. Davies, the Public Analyst, said that he always worked upon the B.P. standard.

Mr. N. G. Goldie, for the defence, contended that there were two standards for cream of tartar,—namely, one for the purified product (obtainable from Pharmacists), and the other for the ordinary commercial article, which could be purchased from grocers for use in cooking and other purposes.

Mr. W. H. Roberts, City Analyst for Liverpool, stated that there was a distinct difference between the two qualities. Genuine cream of tartar was supplied in this case, and the amount of lead was not injurious to health. He would not condemn a sample containing less than 100 parts per million as one likely to cause injury to health.

The Bench held that the inspector had received, not what he had expected, but what he had asked for. As they had been asked by the prosecution to fix a standard, they considered that cream of tartar containing up to 50 parts of lead per million was safe for ordinary commercial purposes.

The case was dismissed.

LEAD IN CREAM OF TARTAR.

It will be generally admitted that the advance in methods of manufacture, as well as in our knowledge of physiological chemistry, makes it desirable that standards of purity for articles used in food should be, at least, maintained—certainly not lowered. The decision of the Birkenhead Bench in a recent case of lead in cream of tartar is regrettable, in that a lax standard of purity is set up for this article as the result of divergence of evidence by two Public Analysts.

In the discussion on Tatlock and Thomson's paper (Analyst, 1908, **33**, 173), C. A. Hill pointed out that 0·002 per cent. was quite a reasonable maximum figure, and that no difficulty was found in obtaining that degree of purity. This is in agreement with the writer's experience in the examination of a large number of trade samples. Even second-grade, 98 per cent. cream of tartar usually only reached 0·002 or 0·0025 per cent. This was 16 years ago, and methods of manufacture are surely not worse to-day.

The standard suggested in Dr. MacFadden's Report to the Local Government Board in 1907 is that arrived at years before by agreement between manufacturers, users and analysts, namely, 0·002 per cent., and this is for the commercial article. It is noticeable that the same figure was adopted in the B.P. 1914 for "purified cream of tartar," so that as far, at least, as lead is concerned, no distinction is to be drawn between the purified article and the commercial product. This is only reasonable, in view of the fact that cream of tartar is used almost exclusively for human consumption, either in food or medicine.

Apart from the merits of any particular case, it is unfortunate that a standard should be set up which differs from the well-known and widely-accepted figures suggested by official reports, without the hearing of evidence from disinterested parties, manufacturers, users and chemists.

H. E. Cox.

The Laboratory,
11, Billiter Square, E.C.3.

GOLDEN SYRUP WARRANTY.

Culver *v.* Sanders.

On January 16th the Divisional Court (the Lord Chief Justice and Justices Avory and Greer) heard an appeal by the respondents against their conviction by the Croydon justices (*cf.* Analyst, 1923, **48**, 544).

Mr. Levy, for the appellants, said that it was admitted that the article sold was not golden syrup, and no doubt contained more glucose and less cane sugar than it should have done. It was sold in tins on which were the words "Amolco, an ideal food for all uses for which sugar is used . . . ; is a nutritious and delicious golden syrup containing a high percentage of sugar."

The tins were supplied from the American packers under a contract contained in a sale note "We have this day sold you 100 cases each containing 36 tins Amolco brand pure cane syrup."

Counsel pointed out that pure cane syrup was a trade name for, and was synonymous with, golden syrup, and he contended that the sale note and label contained a written warranty that the tin contained golden syrup, and that therefore the defendants ought not to have been convicted.

The Court dismissed the appeal without calling on counsel for the respondents. Mr. Justice Avory said that the sale note and label did not agree with each other. The sale note was a sale of tins labelled "Amolco" brand pure cane syrup, and that could not be treated as a warranty that what was being sold was pure golden syrup. As there was no warranty under the Act, the justices were right in the conclusion to which they came.

Department of Scientific and Industrial Research.

REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1922.*

The outstanding event of the year was the beginning of work at the Low Temperature Research Station at Cambridge, and already this station has begun to fulfil its double purpose of acting as an instrument of research for the various Committees of the Board and of taking its part in the general scientific work of the University of Cambridge.

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* Copies of the Report can be obtained from H.M. Stationery Office, Imperial House, Kingsway, W.C.2. Price 1s. 6d. net.

As a result of apples in this country exhibiting "brown heart" disease a small expedition was requested to go to Australia, and left early in 1923 to investigate the gases in ship's holds in which apples were stored.

The several Committees (I–VII) have continued their work during the year, and the Report deals with the results obtained in some detail.

SECTION I.:—THE THEORY OF THE FREEZING OF TISSUES.—An examination into the critical rate of cooling for beef and certain fish was interrupted by the discovery that in the case of eggs preservation does not depend upon the critical rate of cooling being exceeded, but upon the rate falling within certain limits, and these experiments have necessitated a new outlook on the whole question, and have completely upset the accepted theory of freezing of tissues.

SECTION II.:—FISH PRESERVATION COMMITTEE.—Work was chiefly directed to the investigation of certain superficial defects developed by fish on storage with brine freezing, such as the appearance of a central opaque white patch in the eye and a brown colour in the gills. Further points to which attention was directed were the brine freezing of white fish and the effect of the composition of the brine upon the storage properties of the fish.

SECTION III.:—MEAT COMMITTEE.—Work on carbohydrate and nitrogenous autolysis was carried on at "chilling" and cold storage temperatures. The question of the origin of small quantities of succinic acid in muscle is being investigated and a method for the estimation of the succinic acid has been evolved, there being some indication that during storage the quantity of acid rises to a maximum and then falls off. The study of the colour changes developing when meat is frozen in brine was continued and the conditions governing the change of oxyhæmoglobin to methæmoglobin ascertained A full report on work carried out on the conditions governing the growth of bacteria will shortly be ready.

SECTION IV.:—ENGINEERING COMMITTEE.—Two problems received from the Fruit and Vegetables Committee received attention. (1) The question of adequate cooling of stores suitable for the small grower, and (2) the leakage of carbon dioxide through the walls of stores in which the method of gas storage is employed. In connection with these problems the porosity of a large number of materials was tested, and tests are still being carried out. The work on the thermal properties of various insulating materials has been continued and a report for ethyl chloride prepared. The "time lag" of thermometers under cold store conditions has been investigated and the testing of a combined hair hygrometer and dew point apparatus has led to the conclusion that the dew point apparatus is an efficient check on the hair hygrometer (precautions being taken to guard against draughts), and that the latter will keep its calibration for several months to within 2 to 3 per cent., provided changes of temperature and humidity are not too great. Experiments on the relationship of density of packing of slag wool, and heat conductivity established a well-defined minimum value for the thermal conductivity.

SECTION V.:—FRUIT AND VEGETABLE COMMITTEE.—Experiments on the cold storage of home-grown apples were continued; amongst other results the effect of the type of soil in which the trees were grown upon the storage life of the apples was confirmed; the "age of tree" factor was found to be unimportant; oiled wrappers were successfully used; 1° C. was again found to be the most satisfactory cold store temperature. Strawberries and loganberries can be held in cold store in good condition for market for at least 10 days, and in the case of apples in the "gas" store at Histon the average gross sale price after storage was 2·34 times the gross cost of the apples when received for storage. In this store a great variation in the keeping qualities of apples from differen localities was noticeable, and the use of oil wrappers was found to be essential.

Work on the respiration of apples is being directed to correlating the respiration-age relationship and soil effect upon storage life. Knowledge of the various apple diseases has been considerably advanced, and there is evidence that the openings of the minute tubes containing air (utilised in the respiration of the fruit) are the points where many infective diseases begin. The chemistry of ripening continued to receive attention, particularly as regards the sugar content of apple juice, its acidity, the development of pectin on storage, and the parasitic fungi of stored apples.

SECTION VI.:—OILS AND FATS.—Since, with the exception of the α-methyl ether, the methyl ethers of glycerol have not been characterised, and they were needed as reference compounds in the study of the products of fat hydrolysis, their preparation was proceeded with. Work on oleic acid was continued, and attempts were made to synthesise naturally occurring higher unsaturated acids (oleic, tariric) by methods allowing of no ambiguity as to the position of double bonds. At the Lister Institute the study of conditions affecting the formation of fat by the yeast cell has been continued, and information as to the condition in which fat occurs in the vegetable cell has been gained by seeing how far the fat could be removed without hydrolysing the cell substances.

SECTION VII.:—CANNED FOODS COMMITTEE.—The method used for estimating the amines produced by the action of bacteria upon fish depends upon the action of sodium nitrite in acid solution, the titration being carried out in the presence of alcohol. Inorganic phosphate in aqueous extracts of herring was estimated by a preliminary precipitation of protein with dilute hydrochloric acid followed by the magnesium citrate method applied to both filtrate and washings; or by addition of magnesium citrate mixture, solution of the precipitate of magnesium phosphate with part of the protein in hydrochloric acid, addition of alcohol to the extent of 80 per cent., filtration of the re-precipitated protein, and application of the magnesium citrate method to the filtrate and washings.

A preliminary study of the changes occurring during cooking and storing of herrings seems to show that the presence of tin does accelerate the production of simple breakdown products, especially during cooking.

An appendix gives the list of publications during 1922-23, comprising Special Reports and Papers published in Scientific Journals.

D. G. H.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Proteins of Milk-Whey. Their Separation by the Acetone Method. M. Piettre. (*Comptes rend.*, 1924, **178**, 333–335.)—The acetone method, previously applied to blood serum (*Comptes rend.*, **1920**, **170**, **1466**; 1924, **178**, **91**), is employed to separate the proteins of whey as follows: The casein is precipitated in fine flocks by shaking the whey vigorously at about 35° C., and adding, drop by drop, a moderately concentrated solution of commercial rennet. After filtration by means of suction, the whey is treated with 0·7 c.c. of $N/100$ hydrochloric acid per c.c. and is then carefully exhausted with ether without excessive emulsification of the mixture. The clear, decanted liquid is then treated at about 0° C. with 1·35 times its volume of cooled acetone and almost immediately filtered with the aid of the pump. The residue on the filter is moistened and removed to a dish with a volume of water equal to about one-tenth of that of the whey treated. After 12 hours any lumps are crushed and the well-cooled mixture rendered homogeneous by shaking it with ether. Centrifugation then separates a clear, pale liquid, containing the lactalbumin, from the whitish insoluble protein, which may be washed by centrifugation once or twice with water saturated in the cold with ether. The white flocculent matter, occurring to the extent of about 2·3 grms. per litre of milk, resembles the globulins in its physical characteristics and its chemical reactions, although, like casein, it always contains inorganic matter, largely calcium phosphate; in alkaline solution its specific rotation is – 31·2°.

The lactalbumin is purified by either complete or fractional precipitation with 96 per cent. alcohol, 3·2 grms. being obtained per litre of milk; it has $[\alpha]_D -41{\cdot}23^\circ$, and coagulation commences sharply at 67–70° C. and extends to 76–78° C. T. H. P.

Estimation of Alcohol in Bread. T. Sundberg. (*Ann. Falsificat.*, 1923, **181**, 531–534.)—About 200 to 300 grms. of bread are cut into small cubes and immediately placed in a two-litre round-bottomed flask connected with a condenser and standing in a calciumchloride bath. The temperature of the bath is raised to 100°–105° C., and a current of steam is passed through the flask till about 100 c.c. have been collected. Another receiver is placed in position, the calcium chloride solution heated to 110°–115° C., and a further 100 c.c. distilled. The specific gravity of both distillates is determined and the alcohol content deduced from the table of K. Windisch. If the distillates show separation of a thin film of oil, they are filtered through a damp filter-paper. If they are at all acid, they are neutralised with 0·1 *N* sodium hydroxide solution (solid phenolphthalein as indicator), and redistilled through a fractionating column. Any acetaldehyde counts as alcohol, but this error is counteracted by the presence of small amounts of furfuraldehyde. Dry bread contains practically no alcohol, and of the numerous varieties of fresh bread mentioned, Stockholm French rolls contained 0·1 to 0·2 per cent., malt bread 0·2 to 0·5 per cent, and samples of "pain aigrelet de Kalmar" 0·6 per cent. of alcohol by weight. R. F. I.

Use of Essential Oil of Mustard as a Preservative for Wine. P. Malvezin and G. Bidart. (*Ann. Falsificat.*, 1923, **181**, 534.)—The essential oil of black mustard seed (b.pt. 150·7° C.) is soluble in alcohol and ether, but in water only to the extent of 1·16 grms. per litre at 15° C. Its antiseptic power, when used in wines, is such that one c.c. of a one per cent. solution added to 1 litre of wine is efficient, but the amount required varies with certain factors, such as the amounts of sugar and sulphurous acid in the wine, the quantity and vitality of any yeasts present, and the temperature. The souring of red wines, which generally contain no sugar, can be stopped by the addition of 0·8 c.c. of the one per cent. solution above mentioned. Essential oil of mustard is about 200 times as active as sulphurous acid and does not affect the odour or flavour of the treated wines. It does not volatilise from the wine, and therefore does not require to be renewed. It has no action on the colouring matters of red wines, fruit juices, caramel, etc.

It is estimated by heating 5 c.c. of the preservative on a water bath for one hour with 10 c.c. of ammonia solution and 55 c.c. 0·1 *N* silver nitrate solution in a graduated 100 c.c. flask connected with a reflux condenser. The solution is cooled, diluted to the mark, filtered from the precipitate, and the silver estimated volumetrically in 50 c.c. of the filtrate. R. F. I.

Protopectin and some other Constituents of Lemon Peel. R. Sucharipa. (*J. Amer. Chem. Soc.*, 1924, **46**, 145–156.)—The so-called pectins of fruits are of three kinds, *viz.* free pectin, which is water-soluble and of great jelly-making power, protopectin a pectin-cellulose compound insoluble in water, and pectic acid which is the product of the hydrolysis of the other two. Free pectin is the fully methylated ester of pectic acid. Albedo, the white material from lemon peel, after extraction of the essential oils, resins, etc., with petroleum spirit, is composed almost entirely of the three pectins with hemicelluloses and cellulose. After extraction with alcohol and ether, the free pectin in the dry powdered residue is extracted with water, the cellulose is removed by repeated treatment with Schweitzer's reagent, and the residue, after washing out the copper with dilute acetic acid, is then protopectin. It is suggested that in protopectin the cellulose is linked with the pectic acid by carboxyl groups. This complex is split up by heat, the cellulose being replaced by methoxyl groups; this is essentially the ripening process. The heat of the sun hydrolyses the protopectin, forming water-soluble pectin, which renders the whole of the tissue soft. H. E. C.

Estimation of the Extract from Tea. R. E. Andrew. (*J. Assoc. Off. Agric. Chemists*, 1923, **7**, 154–157.)—The following procedure has been favourably reported on by a number of analysts, and is recommended for adoption as the official method for estimating the water-extract in tea: Two grms. of the ground sample are treated with 200 c.c. of hot water in a 500 c.c. measuring flask closed with a rubber stopper, through which passes a glass tube 30 inches long as condenser. The liquid is boiled gently, so that no steam escapes from the tube, for an hour, with occasional rotation of the flask. The cooled liquid is afterwards made up to volume, mixed, and filtered through a dry filter paper, 50 c.c. of the filtrate being evaporated to dryness over a steam-bath and the residue dried in an oven at 100° C. for an hour, cooled, and weighed. T. H. P.

Composition of Commercial Mustard Brans with Special Reference to the Detection of Added Mustard Bran in Prepared Mustard. R. Hertwig and J. I. Palmore. (*J. Assoc. Off. Agric. Chemists*, 1923, **7**, 170–173.)—The figures given below show that the compositions of commercial mustard brans, comparatively free from inside seed materials, differ markedly from those of whole seed; such difference is accentuated by the ratios given. The proposed ratios between nitrogen, crude fibre, total phosphoric acid, calcium oxide, and magnesium oxide should be of valuable assistance in the detection of small quantities of added mustard bran in prepared mustard in the many instances when the crude fibre alone does not show its presence.

The limiting percentages and ratio values, calculated on moisture-free and fat-free material, are: (1) For the seeds: (*a*) Nitrogen, 6·42–8·35; (*b*) crude fibre, 7·20–11·10; (*c*) total P_2O_5, 2·28–4·40; (*d*) CaO, 0·734–1·734; (*e*) MgO, 0·616–1·326. The ratio, (*f*) N: crude fibre, 0·56–1·00; (*g*) P_2O_5: crude fibre, 0·28–0·49; (*h*) MgO: crude fibre, 0·07–0·16; (*i*) CaO: MgO, 0·79–1·71; (*k*) CaO × crude fibre: $P_2O_5 \times N$, 0·26–0·78; (*l*) CaO × crude fibre: $P_2O_5 \times N \times MgO$, 0·21–0·88. (2) For mustard brans, the corresponding values in order have been found to be: (*a*) 2·70–5·92; (*b*) 14·1–29·2; (*c*) 0·40–2·42; (*d*) 0·862–1·910; (*e*) 0·266–0·792; (*f*) 0·09–0·42; (*g*) 0·01–0·17; (*h*) 0·01–0·06; (*i*) 1·2–7·2; (*k*) 1·3–52·3; (*l*) 1·6–19·66. T. H. P.

Salad Dressing. A. E. Paul. (*J. Assoc. Off. Agric. Chemists*, **1923**, **7**, 138–140.)—A salad dressing composed of cottonseed, arachis and olive oils, vinegar, mustard, sugar, salt, egg yolks and boric acid was analysed by four analysts, estimations being made of the total solids, the reducing sugars, before and after inversion, sucrose, total and volatile acids, oil and lecithin-P_2O_5, by the methods given by Lepper (*ibid.*, 1921, **5**, 248). The approximate egg-content was calculated from Jack's figure 0·1316 grm., for the amount of lecithin-P_2O_5 in the yolk of an average egg. The results obtained by the four analysts show that the methods used are quite satisfactory. T. H. P.

Baudouin's Test for Sesame Oil. C. O. Gravenhorst. (*J. Ind. Eng. Chem.*, **1924**, **16**, 47–48.)—The coloration given by genuine sesame oil in the Baudouin test depends, to some extent, on the origin of the oil; the intensity of the coloration is affected considerably by a decrease in the strength of the hydrochloric acid used, the strongest reaction being obtained with an acid of sp. gr. 1·190 to 1·197. The presence of a moderate amount of free chlorine in the acid is without influence on the test. Whilst rancid sesame oil will give a normal reaction with the test, a mixture of sesame oil with arachis oil or butter ceases to give a coloration when it has become badly rancid. If, however, the rancid mixture is washed with warm 5 per cent. sodium hydroxide solution and then dried, it will yield a normal coloration when subjected to the test. W. P. S.

Constituents of Pecan Oil. P. D. Boone. (*J. Ind. Eng. Chem.*, **1924**, **16**, 54–55.)—Pecan oil is obtained from the edible nuts of a hickory tree (*Carya olivoeformis*) growing in Florida, Georgia and Alabama; the oil has a light yellow colour and a pleasant odour and taste, and its characteristics are as follows:—Sp. gr. at 20°/20° C., 0·9118; saponification value, 191·5; acid value, 0·80; iodine value (Hübl), 97·1; refractive index at 20°/20° C., 1·470. The composition of the oil is approximately:—Oleic acid (as glycerides), 80; linolic acid (as glycerides), 16; palmitic and stearic acids, 4 per cent. Phytosterol, m.p. 134° C., is also present. W. P. S.

Glycerides of Lard. C. Amberger and A. Wiesehahn. (*Zeitsch. Unters. Nahr. Genussm.*, **1923, 46,** 276–291, 291–299.)—I. The fatty acids of lard, prepared from different parts of the pig, were first separated in the usual way into solid and liquid portions, and found to consist of stearic acid, 7·8; palmitic acid, 32·2; and oleic acid, 60·0 per cent. Then, in order to find the constitution of the glycerides, their separation was effected by repeated fractional crystallisation. The first stage consisted in solution in warm ether, followed by cooling and separation of the soluble and insoluble portions, which were then fractionally crystallised from alcohol, the progress being followed by the estimation of iodine value and m.pt. These fractions were collected into four distinct groups, which were again separated by dissolving them in acetone and cooling the solution to 0° C., when two layers form, the upper one containing the more unsaturated glycerides. From the constants of the various fractions and comparison of them with those of known glycerides it is deduced that the unsaturated glycerides present in pure lard fat include oleodistearin of m.p. 42° C., oleopalmitostearin of m.p. 41° C., and palmitodiolein, which is a colourless oil at summer temperature, and that triolein may be present, although not in all samples. The percentage composition of fat from the omentum was calculated to be: Palmitodistearin, 3; stearodipalmitin, 2; oleodistearin, 2; oleopalmitostearin, 11; and palmitodiolein, 82.

II. Pure glycerides prepared by known chemical methods, the details of which are given, were compared as to their properties and constants, and those of some of their products of hydrogenation, with the glycerides separated from lard by the foregoing methods. From such comparison it is concluded that the configuration of the glycerides is:—β-palmito-α-α-distearin, β-stearo-α-α-dipalmitin, α-oleo-α-β-distearin, α-palmito-α-β-diolein, and probably β-oleo-α-palmito-α-stearin. H. E. C.

Acids of Beeswax. A. Gascard and G. Damoy. (*Comptes rend.*, **1923, 177,** 1222–1224.)—In view of the confusion existing in the literature with reference to the acids present in beeswax, the authors have investigated this question by saponifying the wax, exhausting the calcium salts of the acids with boiling alcohol, and separating the free acids by fractional crystallisation from alcohol of different strengths at definite temperatures, the solutions being filtered at each temperature. This procedure yields four acids:—(1) Neocerotic acid, $C_{25}H_{50}O_2$, melting at 77·8° C. (*cf.* Marie, *Bull. Soc. Chim.*, 1896, **15,** 510); (2) cerotic acid, $C_{27}H_{54}O_2$, melting at 82·5° C., identical with that furnished by Chinese wax; (3) montanic acid, $C_{29}H_{58}O_2$, melting at 86·8° C., apparently identical with the acid of this name extracted from montan wax (*cf.* Tropsch and Kreutzer, *Brennstoff-Chem.*, 1922, **3,** 49, 177, 193, 212); (4) melissic acid, $C_{31}H_{62}O_2$, melting at 90° C. T. H. P.

Alcohols and Hydrocarbons of Beeswax. A. Gascard and G. Damoy. (*Comptes rend.*, **1923, 177,** 1442–1443.)—By prolonged saponification of beeswax with excess of potassium hydroxide, the authors have obtained the following alcohols and hydrocarbons, corresponding with the acids obtained (preceding abstract). *Alcohols*:—Neoceryl alcohol, $C_{25}H_{52}O$, melting at 75·5° C.; ceryl alcohol, $C_{27}H_{56}O$, melting at 80° C., already found in Chinese wax; montanyl alcohol, $C_{29}H_{60}O$, melting at 84° C., and myricyl alcohol, $C_{31}H_{64}O$, melting at 87° C.; the second and fourth of these alcohols are obtained in largest proportions. *Hydrocarbons*:—Pentacosane, melting at 54–54·5° C.; heptacosane, melting at 59·2–59·5° C.; nonacosane, melting at 63·5° C.; and hentriacontane, melting at 68·4–69° C. T. H. P.

Note on Podophyllin. D. B. Dott. (*Pharm. J.*, 1923, **57**, 661.)—In view of the fact that the resins of *Podophyllum peltatum* and *Podophyllum emodi* have been stated to possess similar pharmacological properties, and both to be soluble in B.P. ammonium hydroxide solution, their behaviour with 25 c.c. of such a solution was examined by macerating 0·25 grm. of each resin for half an hour, filtering, washing the residues with 25 c.c. of water and weighing. In the case of *Podophyllum peltatum* there remained 3·6 per cent. of residue, and of *P. emodi*, 40·4 per cent. Half a grm. of two other samples, when treated, respectively, for 15 minutes each with 15 c.c. of ammonium hydroxide solution diluted with 15 c.c. of water, filtered, and the residues washed with 30 c.c. of water, gave the following amounts of insoluble matter:—*P. peltatum*, 6·0; and *P. emodi*, 47·2 per cent. Treatment with ammonium hydroxide solution thus affords a rapid method for distinguishing the two resins, but an examination of a large number of genuine samples would be necessary before limits of solubility could be definitely stated. On treating 0·5 grm. of each of the two resins for 10 minutes with 10 c.c. of acetone, and washing the filtered residue with a further 10 c.c., 8·2 per cent. of insoluble matter remained in the case of *P. peltatum*, and only 2·0 per cent. in that of *P. emodi*. It would hardly appear likely that substances differing so substantially in their properties would have a similar therapeutic value. D. G. H.

Colour Test for Chloroform and Chloral Hydrate. J. H. Ross. (*J. Biol. Chem.*, 1923, **58**, 641–642.)—The red colour produced on heating chloroform, bromoform, iodoform and chloral hydrate with aqueous sodium hydroxide and pyridine may be used to identify these compounds; and, further, to identify substances which produce these compounds under given conditions and in amounts inferior to 0·005 mgrm. Piperidine and quinoline, when used instead of pyridine, do not produce this colour. The test may apply only to substances which contain the grouping $R{-}CHlg_3$ (Hlg. = halogen). The method is as follows:—Three to 5 c.c. of concentrated (17 to 25 per cent.) sodium hydroxde solution in a test-tube are covered with a layer 2 mm. thick of pyridine. A small quantity of the substance or a drop of the solution to be tested is added, and the contents of the tube are raised to the boiling point, with shaking to avoid bumping. If the colour has not appeared in a few seconds after boiling, the tube is shaken vigorously, and held until the pyridine layer has risen to the surface. The presence of chloroform, etc., colours the pyridine pink or deep red. P. H. P.

Chemical Examination of some Commercial Brands of Carbon Tetrachloride. L. E. Warren. (*Amer. J. Pharm.*, 1923, **95**, 864–874.)—Carbon tetrachloride is now being extensively used for the treatment of hook-worm, and it is important to ascertain the purity of the drug used. Since carbon tetrachloride is usually prepared by chlorinating carbon disulphide, free chlorine and carbon disulphide are likely impurities. The presence of free chlorine, however, is rare, but carbon disulphide, which possesses toxic properties, is frequently present and may be estimated as follows:—Five grms. of the sample are heated beneath a reflux condenser for 30 minutes with 20 c.c. of 0·5 *N* alcoholic potassium hydroxide solution; 50 c.c. of water and 5 c.c. of 20 per cent. alcoholic potassium hydroxide solution are then added, the mixture warmed till the carbon tetrachloride and alcohol have been removed, and an excess of bromine water gradually added. After warming for 15 minutes, an excess of barium chloride solution is added, and the barium sulphate formed filtered off, and estimated in the usual way. ($BaSO_4 \times 0{\cdot}16308 = CS_2$.) Of 9 brands of carbon tetrachloride examined one contained no carbon disulphide, and the amount present in the others varied from 0·015 to 0·27 per cent. The specific gravity at 25°/25° C. varied between 1·5879 and 1·5897, and the boiling points lay between 75·9 and 76·4° C. The Council on Pharmacy and Chemistry for New and Non-Official Remedies require that medicinal carbon tetrachloride should be clear, colourless and mobile, of specific gravity not less than 1·588 at 25° C., and that it should boil between 76 and 77° C. On shaking 10 c.c. with 10 c.c. of water the aqueous layer should be neutral to litmus, give no opalescence with silver nitrate solution (*chlorides*), and, on addition of a few drops of potassium iodide solution, should not be coloured violet in 5 minutes (*free chlorine*). On warming 10 c.c. of medicinal carbon tetrachloride with 10 c.c. of 25 per cent. potassium hydroxide solution no yellow or brown colour should develop (*aldehydes*), and on mixing 10 c.c. with 10 c.c. of sulphuric acid and shaking occasionally for 5 minutes, not more than a barely perceptible trace of colour should develop in either layer (*organic impurities*). On evaporating 25 c.c. almost to dryness and leaving the remainder to evaporate spontaneously, the residue, if any, should be odourless and should not weigh, on drying, more than 0·001 grm. Not more than 0·1 per cent. of carbon disulphide, estimated as described above, should be present.

D. G. H.

Biochemical, Bacteriological, etc.

Solubility of Copper in Milk. F. E. Rice and J. Miscall. (*Inter. Rev. Sci. Pract. Agric.*, **1923, 1,** 1095–1096.)—This work was carried out to find to what extent milk can act as a solvent of copper, since this metal was found in cows' milk. Some milk, after being left in contact with a strip of pure copper in a glass vessel, was evaporated to dryness, the residue was burnt, and the ash taken up with nitric acid. The copper that had passed into solution was estimated either electrolytically or by the ethyl-xantho-potassic method. If copper vessels are used in dairy work, they should be closed, since air and oxygen enormously increase the corrosive action of milk on copper; carbon dioxide does not affect it. A film of oxide on the copper also increases the solubility; dairy utensils should be clean and not disinfected with chlorine compounds. Boiling appears to destroy or transform the factor upon which the solvent action of milk depends, and thus copper is least easily attacked by boiling milk. The addition of sugar (to about 18 per cent.) does not increase the action, nor does a slight amount of acid. Experiments with cream and whey showed that the metal is chiefly dissolved by the water. Theoretically and experimentally it appears that copper in solution in milk can be displaced by tin; therefore, condensed or evaporated milk kept in iron receptacles lined with tin will contain less copper than the original product as made in unlined copper utensils. P. H. P.

Action of Alkaline Compounds on Insulin. E. J. Witzemann and L. Livshis. (*J. Biol. Chem.*, **1923**, **58**, 463–474.)—Tables of results are given showing the lability of insulin in the presence of alkali. Preparations were injected into rabbits that had been made to fast during the 18 hours previous to the injection. The blood sugar concentrations were determined before, and again 2 hours after, injection, unless the rabbits suffered convulsions, in which case blood was taken when convulsions were first observed. Results show that insulin is more or less completely inactivated at room temperature by 0·5 or 0·7 *N* ammonium hydroxide in the course of some days, and that its original activity is usually quickly restored on acidifying the ammoniacal solution with hydrochloric acid. Sodium and potassium hydroxides, even at 0·1 *N* concentration, inactivate insulin irreversibly at room temperature. Sodium carbonate and bicarbonate and disodium phosphate had hardly any action upon insulin. Exploratory experiments on the effects of mixed alkalis upon insulin indicate that the above reactions are subject to secondary accelerative and retarding effects depending on the mixture used. The nature of the chemical action of alkaline compounds and of ammonium hydroxide upon insulin are discussed. The reversible effect of ammonium hydroxide suggests a tautomeric re-arrangement, but nothing definite can yet be said as to the nature of the groups that would be involved in this re-arrangement. P. H. P.

Vitamin *C* in Canned Foods. W. H. Eddy and E. F. Kohman. (*J. Ind. Eng. Chem.*, **1924**, **16**, 52–53.)—To determine the extent to which vitamin *C* is destroyed in the process of canning foods, fresh cabbages were packed in tins, the tins were then filled with boiling water, the air was exhausted, and the tins sealed and heated at 100° to 126° C. for periods varying from fifteen to sixty minutes. The results of subsequent feeding experiments with this canned cabbage indicated that the destruction of vitamin *C* is dependent on some factor other than temperature and time of heating and that, in any case, the process is less destructive than cooking in open vessels. Protection against scurvy was afforded by 4 or 5 grms. of the canned products when given to guinea-pigs, and, with smaller doses, the product heated at 115° C. was more effective than that which had been heated at 100° C. for the same length of time. W. P. S.

Fat-Soluble Vitamins, XVI. H. Steenbock, J. H. Jones and E. B. Hart. (*J. Biol. Chem.*, **1923, 58,** 383–393.)—The authors describe the preparation of ether extracts of saponified cod liver oil. When rats were fed with this material it was found that the vitamin *A* content had not been sacrificed, nor had the anti-rachitic vitamin been destroyed by the saponification and extraction. Some dogs were kept on a basal ration and, after several weeks, deformation occurred, and they had difficulty in walking, owing to maintenance of weight with growth in size and gradual decalcification of the skeleton putting great strain upon the skeleton. This deformation usually occurs with animals afflicted with rickets late in the course of the disease. Where the basal ration was supplemented with cod liver oil, saponified or untreated, growth was very uniform, and continued so until the experiment was terminated. Figures are shown of sections, taken through the thorax to include the fifth rib, at the end of the experiment. The sections of the dogs fed on the basal ration alone show several partly repaired fractures and enlarged costochondral junctures. Analyses for ash carried out showed that the growth itself of these dogs had actually been interfered with. A lowering also of the calcium and phosphorus content of the blood occurred in the basal animals. These dogs had severe tetanic convulsions, but these have been observed in dogs afflicted with rickets that had a normal calcium content of the blood. Apparently a decreased calcium content cannot *per se* be the cause of these convulsions. Therefore, from the standpoint of growth, calcification of bones and maintenance of normal calcium and phosphorus content of the blood, the ether extract of saponified cod liver oil is as efficacious as the untreated oil. P. H. P.

Bacteriological Examination of Ground Beef, I. R. E. Hoffstadt. (*Amer. J. Hygiene*, **1924, 4,** 33–42).—Different samples of meat were purchased from public markets (open and closed stalls) and from shops. Some were ground for the purchaser and others were ready for sale. They were examined by St John's method as cited by Tanner (*Bacteriology and Mycology of Food.* Chapter XIII.), which was modified by using the mechanical shaker. The meat was shaken with sterile glass beads in sterile water and plated in ordinary media. The bacterial count of market samples showed a wider range than that of shop samples. The bacterial count of all samples showed much variation in relation to sanitary conditions, *i.e.* the condition of floor, counter and ice-boxes, and the organoleptic test. The total aerobic count or count of dextrose fermenters is unreliable as a standard for meat analysis. The presence of an aerobic form or group cannot be taken as an index to the freshness of the meat. There was no relation between initial counts and counts in subsequent spoilage of samples, and there was no relation betweenthe percent-age of proteolytic aerobes and the type of spoilage. Colour and odour were two of the points considered when grading the meat as good or poor. P. H. P.

Bacteriological Examination of Ground Beef, II. R. E. Hoffstadt. (*Amer. J. Hygiene*, **1924, 4,** 43–51.)—From the results obtained in this work it may be concluded that the presence of proteolytic anaerobes affords a definite indication as to the keeping qualities of meat. The technique described for indicating their presence can be adapted easily for laboratory use. Anaerobiosis can be successfully obtained by layering with vaseline and the McIntosh and Fildes jar (*Lancet*, **1916**, **1**, 768) for incubation can be dispensed with. The method used in isolating the organisms is given, with full descriptions of six different types of anaerobic species represented by 110 cultures which were identified, *viz.*, 2 saccharolytic, 2 moderately proteolytic and 2 strongly proteolytic types. A definite type of spoilage indicates the presence of each group. There is no relation between the anaerobe present, the original organoleptic test, the sanitary conditions and the aerobic count, although each type of spoilage represents a special type of anaerobe.

P. H. P.

Bile Acids in Urine. C. L. A. Schmidt and J. A. Merrill. (*J. Biol. Chem.*, 1923, **58**, 601–609.)—Results of repeated investigations on this subject are not in harmony. It is thought that bile acids are eliminated in the urine only in small quantities, and the question is raised as to the fate of these substances. To study the subject an accurate method for the quantitative estimation of the bile acids in urine is a necessary pre-requisite. The principle of the method of Foster and Hooper (*J. Biol. Chem.*, 1919; **38**, 355), modified by Schmidt and Dart (*J. Biol. Chem.*, 1920–21, **45**, 415) in conjunction with an observation made by Tengström (*Z. Physiol. Chem.*, 1904, **41**, 210), has been used by the authors for their estimations. Their results indicate a recovery of 97 per cent. of the bile acid nitrogen from ox bile by precipitation of the bile acids by the addition of magnesium sulphate. They added a solution of the bile acids to urine and carried out experiments to determine the recovery of the acids. Whole human and ox bile were added in varying amounts to specimens of urine, and always more than 90 per cent. of the bile acids were recovered. They found that hippuric acid does not interfere with the estimations carried out by their method. An allowance for an error of 5 per cent. in the estimations should be made. Details and precautions are given. The bile acid output in the urine was estimated in some cases of jaundice; the values were extremely small. There was no constancy in the amounts eliminated by a particular person. The authors do not consider that the amounts in the urine represent the total production of the bile acids in the system. P. H. P.

Organic Analysis.

Refraction Method for Estimating Oil in Paraffin Wax. R. E. Wilson and R. E. Wilkin. (*J. Ind. Eng. Chem.*, 1924, **16**, 9–12.)—Five grms. of the paraffin wax are dissolved in 75 c.c. of ethylene dichloride, the solution is cooled to −18° C. and filtered at this temperature. The insoluble wax is washed with 75 c.c. of ethylene dichloride, cooled previously to −18° C., and the filtrate and washings are evaporated. The residue thus obtained, containing all the oil and a trace of wax, is transferred to a weighing bottle with the aid of a small quantity of ethylene dichloride, and the solvent is evaporated completely. A known quantity (about 0·2 c.c.) of a mixture of "mineral seal" and "ligature" oils is then added to dissolve the wax, and the refractive index of the solution is determined at 25° C. Reference to a graph gives the percentage of oil in this solution and the quantity in the original wax is calculated. The mixture of mineral seal and ligature oils is prepared by mixing 45 per cent. of the former with 55 per cent. of the latter; the mixture has the same refractive index (1·4470) as paraffin wax, and any change in this value is due to the oil present in the residue. Six samples of refined paraffin wax examined contained from 0·08 to 0·28 per cent. of oil.

W. P. S.

Composition of Whale Oil. C. H. Milligan, C. A. Knuth and A. S. Richardson. (*J. Amer. Chem. Soc.*, 1924, **46**, 157–166.)—The composition of whale oil has been investigated by separating the solid and liquid fatty acids by Twitchell's modification of the lead salt and ether method (ANALYST, **1921, 46, 466**), esterifying the separated acids, and then fractionally distilling the methyl esters. From the fractionation it is calculated that the esters of the solid acids were composed of myristic acid, 13·6; palmitic, 68·0; stearic, 16·8; and higher unsaturated acids, 1·6 per cent. The composition of the esters of the liquid acids corresponded to the following percentages:

Carbon content of fatty acid.	Per Cent.	Carbon content of fatty acid.	Per Cent.
14	2·7	20	20·2
16	15·4	22	15·4
18	42·7	24	2·8
		Unsaponifiable	0·7

From this and other data the approximate composition of whale oil fatty acids is calculated to be:

	Per Cent.		Per Cent.
C_{14}, Myristic	4·5	C_{20}, Unsaturated	16
C_{16}, Palmitic	11·5	C_{22}, Unsaturated	10
Palmitoleic	17·0	C_{24}, Unsaturated	1·5
C_{18}, Stearic	2·5	Unsaponifiable	0·7
Unsaturated (nearly all oleic)	36·5		

It is remarked that the mean molecular weight of the raw fatty acids was 277; complete hydrogenation should theoretically increase this only to 279·7, but in practice it was increased to 285·5 by hydrogenation at 217° C.

H. E. C.

Action of Catalysts with Non-drying Oils. A. G. Hill. (*J. Ind. Eng. Chem.*, **1924, 16**, 23–24.)—The presence of small quantities of iron salts in olive oil or "red oil" (commercial oleic acid) causes the oils to become so heated that they may be dangerous in textile factories. Similar effects are also produced by sodium, magnesium, zinc, and other salts, the most energetic action being produced by those metals of which the lower oxide is more stable than the higher oxide. A method of testing oils which may contain traces of metals consists, briefly, in mixing 14 grms. of the oil with 7 grms. of ordinary bleached cotton wadding, and packing the mixture in a wire-gauze cylinder which is placed in a cylindrical copper tube surrounded by a hot water jacket. A current of air is drawn through the apparatus, and water in the jacket is kept boiling. A thermometer is inserted in the oil-wadding mixture, and any oil which shows a temperature exceeding 100° C. after one hour's heating, or 200° C. after two hours, should be regarded as dangerous and liable to produce spontaneous combustion. Of the metallic salts investigated, only those of aluminium, which forms but a single oxide, failed to act as catalysts in the reaction. In the case of zinc, the second oxide has been discovered comparatively recently. (*Cf.* Mackey, *J. Soc. Chem. Ind.*, 1896, **15**, 90.) W. P. S.

Interaction of Glucose and Phenylhydrazine. E. Knecht and F. P. Thompson. (*J. Chem. Soc.*, 1924, **125**, 222–226.)—Quantitative study of this reaction showed a maximum yield of phenylglucosazone of 84 per cent.; this is obtained when 3·6 grms. of glucose, 6·48 grms. of phenylhydrazine and 20 c.c. of glacial acetic acid in a total volume of 100 c.c. are heated on the water bath for 3 hours. Analysis of the resulting mixture shows that the reaction proceeds in accordance with the empirical equation:—

$$C_6H_{12}O_6+3NHPh.NH_2 \longrightarrow C_6H_{10}O_4(:N.NHPh)_2+NH_2Ph+NH_3+2H_2O.$$

For the analysis the uncombined phenylhydrazine in the mixture was estimated by collecting the nitrogen liberated on boiling with Fehling's solution (Strache, *Monatsh.*, 1892, **13**, 299), and the ammonia by distillation and titration with standard hydrochloric acid, litmus being used as indicator, because with it aniline salts react as free acid. H. E. C.

Analysis of Naphthalenesulphonic Acids and Naphthalene. W. S. Calcott, F. L. English and F. B. Downing. (*J. Ind. Eng. Chem.*, 1924, **16**, 27–30.)—In the methods described the naphthalene residue is estimated by oxidising the sulphonic acids to derivatives of phthalic acid by means of an excess of vanadic acid dissolved in 70 per cent. sulphuric acid; the naphthalene is then calculated from the amount of potassium permanganate required to re-oxidise the reduced vanadium. Each molecule of sulphonic acid requires (for oxidation to sulphophthalic acids) nine atoms of oxygen, irrespective of the number and arrangement of the sulphonic acid groups. The sulphonic acid groups may be estimated by boiling a mixture of the free acids and sulphuric acid with barium carbonate, which converts the sulphuric acid into barium sulphate and the sulphonic acids into soluble barium sulphonates; the mixture is filtered and the barium in the filtrate is precipitated with dilute sulphuric acid. The resulting barium sulphate is collected and weighed, and the sulphonic acid sulphur is calculated from the result obtained. The relation between the naphthalene residue and the sulphonic acid sulphur gives the degree of sulphonation in the sample under examination. For the estimation of naphthalene in crude naphthalene, 12 grms. of the sample are placed, together with 100 c.c. of 5 per cent. sodium hydroxide solution, in a round-bottomed 500 c.c. flask which is connected with a similar flask containing 100 c.c. of 10 per cent. sulphuric acid; this second flask is, in turn, connected with a 1 litre receiver, over which a stream of cold water is run, and with a 500 c.c. receiver surrounded by an ice bath. The mixture is steam-distilled for about twenty minutes, the distillation flask and the sulphuric acid flask being heated meanwhile to maintain the volumes of the solutions constant; the first receiver is then removed and another substituted, and the distillation is continued until nothing but oil separates in the receiver or until the receiver fills with a white acrid-smelling vapour. The naphthalene in the receivers is collected on a filter, the receivers are rinsed with 10 c.c. of alcohol; this is diluted with 200 c.c. of water and also passed through the filter; the naphthalene is washed with water from the filter into a beaker, the lumps are crushed, and the substance is again collected on a filter and pressed to remove as much water as possible. The naphthalene is then transferred to a thick-walled pyrex tube, of 110 c.c. capacity, 25 c.c. of concentrated sulphuric acid and 30 c.c. of 20 per cent. "oleum" are added, and the tube is closed tightly with a rubber stopper and heated at 100° C. for one hour. After cooling, the mixture is diluted with 250 c.c. of water, boiled for twenty minutes to expel sulphur dioxide, cooled, and diluted to 1 litre. Twenty-five c.c. of this solution are then oxidised with vanadic acid as described previously.

A method for the estimation of water in naphthalene depends on the change in the critical solution temperature of a standard toluene-water mixture after the addition of the moist sample. W. P. S.

Estimation of Ferrocyanides. W. M. Cumming. (*J. Chem. Soc.*, 1924, **125**, 240–243.)—The estimation of ferrocyanides by precipitation with copper or iron salts is unreliable, different results being obtained according as sodium, potassium or calcium ferrocyanide is used. A method which has been found to give accurate results with all three is based on the precipitation of benzidine hydroferrocyanide, $3C_{12}H_{12}N_2H_4FeC_6N_6.H_2O$, by benzidine hydrochloride in neutral solutions of the ferrocyanides. The method may be applied gravimetrically or volumetrically; in the former case the precipitate is washed, dried, and ignited to Fe_2O_3, the factors 4·599, 3·799, and 3·649 being used to convert Fe_2O_3 into anhydrous potassium, sodium, or calcium ferrocyanide respectively. (The true formula of the crystalline form of the last-named salt includes 11 H_2O not 11½ or 12 as quoted in text books.) The volumetric modification of the above process consists in titrating the ferrocyanide with a benzidine solution of known strength, using as indicator bromine water just decolorised by sodium carbonate and applied as follows:—When the reaction is nearly complete a drop of the mixture is spotted on filter paper, and a drop of the bromine water placed near by so that, on spreading, it touches the other ring; a yellow coloration at the juncture denotes the end-point. It is important to note that this colour is produced at the edge, as at the centre there will always be a yellow colour, owing to decomposition of the precipitate in the air. A satisfactory end-point cannot be obtained on titration in the reverse direction.

H. E. C.

Inorganic Analysis.

Use of Hypophosphorous Acid in Gravimetric Analysis. L. Moser and M. Niessner. (*Zeitsch. anal. Chem.*, 1923, **63**, 240–252.)—*Mercury.* Solutions of mercuric chloride free from nitric acid are treated with an excess of 0·5 molar solution of hypophosphorous acid and 10 c.c. of strong hydrochloric acid, and heated on the waterbath. The metallic precipitate is left to settle, filtered off on tared paper, washed once with dilute hydrochloric acid, then with hot water, alcohol, and ether, dried at 30° C., and weighed in a closed weighing-bottle. Mercurous chloride and sulphate are suspended in water and heated with hypophosphorous acid; the nitrate must be reduced without addition of hydrochloric acid, otherwise some metal would be re-dissolved on heating. Mercury may be separated in this manner from iron, lead, cadmium, and zinc. *Gold.*—The chloride solution is treated with 2 grms. of sodium chloride, heated to incipient boiling, then treated with 0·25 molar hypophosphorous acid. The hot solution is allowed to stand till quite clear before filtration, addition of a little filter pulp being recommended. The precipitate is washed with one per cent. acetic acid till quite free from chlorine, and ignited. *Palladium.*—The feebly acid, boiling chloride solution is treated with an excess of the reducing agent and kept gently boiling for 5 minutes in the covered beaker. When flocculation is complete the hot liquid is filtered, and the precipitate washed with hot water and ignited. The estimation of mercury, gold, and palladium is very accurate. *Platinum* is not precipitated, and may be separated quantitatively from gold; the results are accurate with widely varying proportions of the two metals. On the other hand, the separation of platinum from palladium cannot be accomplished by means of hypophosphorous acid, as palladium catalytically induces the precipitation of platinum. The estimation of *copper* is unreliable; *bismuth* nitrate solution yields a precipitate consisting largely of phosphate, whilst a chloride solution is incompletely precipitated. W. R. S.

Volumetric Estimation of Antimony by means of Permanganate. O. Collenberg and G. Bakke. (*Zeitsch. anal. Chem.*, 1923, **63**, 229–240.)—This method was submitted to a critical investigation; it was found to give correct results, equal to those of the bromate method, provided the acidity is kept within the limits of 10 to 19 c.c. of hydrochloric acid (sp. gr. 1·19) per 100 c.c. of solution. The permanganate solution is standardised against sodium oxalate. Agreement with gravimetric methods is attained only when the new atomic weight—Sb=121·8—is used in the calculation (*cf.* ANALYST, 1924, 49). Petriccioli and Reuter's modification (dilution of the antimony solution with water until oxychloride is precipitated and solution of the precipitate in a minimum of hydrochloric acid, previous to titration) gives negative errors of 1·2 to 1·5 per cent.

W. R. S.

Interference of Cobalt in the Bismuthate Method for Manganese. G. E. F. Lundell. (*J. Amer. Chem. Soc.*, 1923, **45**, 2600–2603.)—Cobaltous salts are oxidised by sodium bismuthate, and the oxidised compound reacts with both ferrous sulphate and permanganate. The action on the latter does not start until some permanganate has been decomposed, after which the reaction proceeds rapidly; the action on the ferrous salt is much slower. The net result is that a low manganese value is obtained. Manganese can be estimated satisfactorily in presence of cobalt by oxidation with persulphate in acid solution, followed by titration with sodium arsenite. W. R. S.

Estimation of Small Amounts of Molybdenum. Application to Ammonium Phosphomolybdate for the Indirect Titration of Phosphorus. A. Vila. (*Comptes rend.*, 1923, 177, 1219–1221.)—Phosphorus may be rapidly estimated, even in amounts below 0·1 mgrm., by estimating volumetrically the molybdenum in ammonium phosphomolybdate. This, or other molybdenum compound, is reduced by means of hydrogen, this reaction yielding either metallic molybdenum or its dioxide, which, in presence of excess of molybdic acid, is converted quantitatively into the blue saline oxide, MoO_2, $4MoO_3$. The ammoniacal solution of molybdic acid or ammonium phosphomolybdate is evaporated to dryness in a transparent silica test-tube; the deposit should cover not more than one-third of the length of the tube. A stream of pure hydrogen is passed through the tube, which is closed and introduced into an electric furnace heated to about 700° C. After the lapse of 15 minutes, the tube is withdrawn and allowed to cool with the hydrogen passing. For converting the residual metal or dioxide into the blue oxide, use is made of a solution prepared by dissolving 5 grms. of pure molybdic acid in 10 c.c. of 60 per cent. phosphoric acid, decolorising the solution exactly with potassium permanganate, and making up to 50 c.c. with water. This reagent is introduced in quantities of 2 c.c. into the tube, which is carefully heated, the molybdenum blue at first formed giving place to a more or less deep green syrupy liquid. This is allowed to cool and is then treated with water and boiled, the resulting molybdenum blue being titrated with $N/400$ potassium permanganate solution until the liquid is completely decolorised: 1 c.c. of this permanganate corresponds with 0·200 mgrm. Mo, 0·300 mgrm. MoO_3, 0·00538 mgrm. P, or 0·0123 mgrm. P_2O_5. T. H. P.

Volumetric Estimation of Titanium. G. E. F. Lundell and H. B. Knowles. (*J. Amer. Chem. Soc.*, 1923, 45, 2620–2623.)—The authors find that, contrary to the generally accepted idea, solutions of titanic sulphate are easily and quantitatively reduced to the trivalent stage by the use of Jones' reductor (19 mm. bore, 43 cm. amalgamated zinc-column). The reductor was washed immediately before use with dilute sulphuric acid, and the receiver emptied and rinsed and filled with ferric sulphate solution (from 3 to 5 times in excess of the quantity required for the oxidation). There were passed through the reductor 25 to 50 c.c. of 3 to 5 per cent. sulphuric acid, 150 c.c. of titanium solution in dilute acid of the same concentration, another 100 c.c. of dilute acid, and, finally, 100 c.c. of water. The reduction was effected at a speed of 100 c.c. per minute. The reduced iron was titrated with 0·1 N permanganate standardised against sodium oxalate. The errors averaged not more than one drop of standard solution. The reduction can be done at any temperature between 25° and 100° C., and it is unnecessary to expel air from the solutions used. W. R. S.

New Reagent for Potassium. H. and W. Davies. (*J. Chem. Soc.*, 1923, **124**, 2976–2982.)—Sodium 6-chloro-5-nitro-*m*-toluenesulphonate is proposed as a reagent for potassium. The solubility of the potassium salt, which forms characteristic voluminous scales, is 0·235 grm. in 100 grms. of water at 0°, and about 0·40 at 20° C. The test can be carried out in cold, strongly alkaline or in dilute mineral acid solution, and potassium can be detected in presence of large quantities of sodium and magnesium. Ammonium must be previously removed by boiling with sodium hydroxide. The reagent will detect one part of potassium in about 2500 of water. The reaction can be applied to quantitative analysis; the results of the analysis of potassium salts were within 0·5 per cent. of theory. The reaction is carried out in aqueous solution dilute enough to allow the potassium sulphonate compound to dissolve completely at boiling heat; the precipitate formed on cooling and standing is washed with a little water, and weighed; a considerable excess of reagent and very small volume of solution are aimed at. Sulphates and moderate amounts of aluminium, sodium, and magnesium, are not detrimental; but alkaline earths interfere, and must be eliminated. The potassium factor is 0·1347. W. R. S.

Detection of Halogens. W. B. S. Bishop. (*J. Soc. Chem. Ind.*, 1924, **43**, 23T.)—In testing for the presence of ethyl iodide in ether a piece of well oxidised copper gauze of such a size as to absorb about one c.c. is dipped into it. The gauze is then placed in a Bunsen flame, which becomes green if halogen is present. This test was found to detect 0·00004 grm. of iodine in one c.c. of ether, and is said to be more delicate than either that of Rübke or Lassaign. It will also detect 0·0001 grm. of bromine in one c.c. of ether. R. F. I.

Apparatus.

Substitute for Platinum in Electrolysis. M. Bleesen. (*Zeitsch. anal. Chem.*, 1923, **63**, 209–228.)—The author submits the results of test analyses in which cathodes of Borchers' alloy were used (Ni, 65–60; Cr, 30–35; Mo, 2–5; Ag, 0·2–1 per cent.). This alloy is rendered passive by immersion in strong nitric acid; the surface is made dull by sand-blasting so that adherent deposits are obtained. The best form of cathode is wire-netting or gauze. The deposit is removed from the cathode by a suitable acid in the cold; the loss in weight of the cathode hardly exceeded 0·0004 grm., and was generally smaller. The results submitted are satisfactory. W. R. S.

Reviews.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. Fifth Edition, Vol. I. Pp. 796. London: J. & A. Churchill. 1924. Price 30s. net.

The appearance of the first volume of a new edition of *Allen's Commercial Organic Analysis* (on this occasion the fifth), is an event. There is no other book in the English language that covers the ground covered by this book, or, for which, it may be added, analysts have so great an affection. It must be allowed that there is no chemist capable, by himself, of covering this large ground adequately, and that this is so is a compliment to the activities of chemists, not a slight on their industry. Consequently, this edition, as was the case with the last, is written by a number of experts in the various sections concerned, under the general editorship of S. S. Sadtler and E. C. Lathrop, as American editors, and C. A. Mitchell, as English editor.

The volume now under review (Vol. I.) deals with the same matters as did the corresponding volume of the 4th edition, published fifteen years ago.

The first section, named "Introduction," is word for word the same as it was in the previous edition (even to the mis-spelling of carbonyl chloride on p. 84); the only addition is a footnote concerning the xylene and petroleum distillation methods for the estimation of water content.

That this section has not been re-written is much to be regretted, for, during the past fifteen years, a number of important advances have been made in physical apparatus and the application of physical means to analysis, and, even when the last edition was published, this section was not particularly serviceable. Colorimeters and immersion refractometers of extended range fail to gain recognition, and there is no reference to hydrogen ion determination or to the large range of modern indicators and the use to which they can be put. Solvents would apparently stand in the same position as they did twenty years ago, which is far from the case.

If reasons of space have led to serious omissions from this section (the volume shews an increase of 200 pages over that of the 4th edition), the section could have been remodelled in order to make room for more useful matter. The chemist who uses "Allen" does not require minute directions to "turn a knob T," and so forth, in the use of a polarimeter, and a page devoted to the use, and a picture, of a separating funnel is too ample an allowance. There is much in this section, in fact, suitable only for a junior student, whilst much that is wanted by a practising chemist is omitted.

Later sections have, to a great extent, been re-written, and in them have been incorporated the later information and data appearing in the Supplementary Volume (Vol. IX.) of the last edition.

The section on the alcohols, revised by L. M. Burghart, has been extended, and deals mainly with ethyl and methyl alcohols. Acetone is included in this section, and certain higher aliphatic alcohols are briefly treated. The ethyl alcohol tables are those of the American Bureau of Standards.

It is agreeable to note that sections on Malt and Malt Liquors, on Wines, Cider, and Potable Spirits, are still permissible in "Allen." These are by J. L. Baker and L. Eynon respectively. They are largely re-written and include much new matter, new, that is, to this book. The former author contributes much from his own work. Particular wines and spirits are but lightly dealt with, this section being devoted chiefly to analytical methods of general applicability. A "British Government Laboratory Method" for the estimation of higher alcohols in spirits is given without any reference.

A short section on yeast by E. Schlichting follows.

Neutral alcoholic derivatives form another section, by H. Leffmann. Aldehydes, ether, esters, chloroform, chloral and so forth, are herein treated.

The important section on sugars has been revised by L. Eynon; sugars and some sugar products are dealt with at length, and glucosides, urine analysis as regards sugar, and pentoses receive a short treatment.

The starch section is by T. H. Pope, and deals with starches, cellulose, gums and cereals. Considering the importance of wheaten flour, the space given to it seems inadequate, especially as the flour industry is becoming so largely a chemical one.

E. Sutermeister contributes a section on paper and wood pulp, and the volume closes with a section on aliphatic acids by H. Schlatter. The acids herein treated comprise acetic acid (including vinegar), and certain homologues, oxalic, succinic, citric, tartaric and malic acids.

The index is, perhaps, adequate, though barely so; it is certainly not a generous one. This fault is partly compensated for by the fact that each section, in a way, provides its own index by means of headings in distinctive type. Short bibliographies are supplied at the end of each section.

A perusal of this volume leads to the conclusion that, in most respects, it is a better production, and a more useful one, than the corresponding volume in the last edition. Criticism, no doubt, there will be, criticisms which are partly an index of the great interest that this book has for English analysts. In these days of complexity it is a matter of difficulty to make a judicious selection of the matter that should be included or excluded, a difficulty that is increased by the endeavour to provide for the needs of analyst in both America and England. Commercial organic analysis, as distinct from "pure" chemistry, has often to be approached from different points of view in the two countries. English readers have cause for regret that this book is no longer an English production in its entirety, but, with that reservation, will welcome this new edition and be grateful for the enterprise that has led to it.

E. HINKS.

THE DETERMINATION OF HYDROGEN IONS. By W. MANSFIELD CLARK, M.A., Ph.D., Baltimore, U.S.A. Williams & Wilkins Company. Second Edition. 1923. Price $5.50.

Since the publication of the first edition of this book (reviewed in THE ANALYST, 1921, **46**, 70), it has become generally recognised that hydrogen ion concentration is an important factor in nearly all chemical reactions, and that, most particularly in the biochemical field, it is as essential that the P_H value should be controlled as that the temperature should be controlled. An increasing number of chemists have found it necessary to make themselves familiar with the fundamental theory of the subject, and to employ one or other of the methods of determination. For this purpose chemists cannot do better than go to this book, which gives a comprehensive description of the theory of hydrogen ion concentration, and treats fully of indicator and electrometric methods of determination. The theoretical part is intelligible to the ordinary reader, considering the present state of flux of the ion question. The new concept of hydrogen ion "activity" put forward by Lewis is mentioned, but the author wisely has made no attempt to introduce this into his general treatment of the subject—a decision which will be welcomed by the ordinary worker, who has no wish to change his standards of reference until something more definite has evolved out of the present confusion. The great expansion in the literature dealing with hydrogen ion concentration is shown by a comparison of the excellent bibliography at the end of the book with that in the first edition, and some idea of the width of the field in which hydrogen ion concentration has been studied may be gained from the chapter on "Applications." This expansion has necessitated a considerable increase in the size of the second edition, which now contains 480 pages as compared with 317 in the first edition. Many chapters have been practically re-written and new ones have been included.

One is glad to see the colour chart of indicators in its original form. Taking into account the difficulties in printing such a chart, it is remarkably well done, and of great value for rough indicator determinations of P_H in the laboratory.

The book is admirably printed, and the reviewer has not observed a misprint. It remains the best book in the English language dealing with hydrogen ion concentration.

NORMAN EVERS.

A TESTED METHOD OF LABORATORY ORGANISATION. By SEYMOUR PILE, M.A. (Cantab.), and REGINALD G. JOHNSTON. Pp. 96. London: H. F. & G. Witherby. Price 7s 6d.

Organisation, as applied to scientific work, has hitherto received but little attention in this country, and a brief account of a laboratory which had to be designed, furnished, staffed and organised, at express speed and during one of the most critical years of the war, should be both interesting and informing to the reader.

The writers, however, do not limit their attention to the merely mechanical details of equipment, methods of work and financial problems which arise in connection with them. They treat also the psychological side of the chemist, the kind of being he is, or ought to be, the influences by which he is moulded, his virtues and failings, his relations to his colleagues, and the difficulties with which he has to contend when in contact with the lay mind.

After an introduction and preface, there follow four chapters dealing with the laboratory, the personnel, the treatment of the sample, and the methods of costing, after which, in three final sections, the writers "let themselves go" and discourse generally on, to use their own words, "the scientist in relation to this, that and the other."

All who own or use a laboratory cannot fail to be interested in the ideas which Messrs. Pile and Johnston have set forth, whether they agree or, as is equally likely, do not agree, as to details. Such points as the use of acid-proof paints, methods of ventilation and fume cupboards, sinks and drains, the width of benches and the illumination of balances, are all matters of interest and of contention.

The personnel of a large laboratory is a subject which gives rise to many problems which have hitherto received but scant attention. Here, too, the authors have ideas of their own which cannot be accused of being either academic or conventional.

Perhaps one of their most revolutionary suggestions is that the members of the staff should be of equal social standing, and this involves the abolition of the "bottle washer" and the "laboratory boy," who, say the authors, "break more glassware than their salaries will pay for." "The staff should clean up its own apparatus."

Greatly daring, they go further, and suggest that "all special knowledge," after a good secondary education, can be acquired in the laboratory and, while they do not minimise the value of academic training and degrees, they consider that the special training for the industrial scientific worker should be given in the works' laboratory itself.

It is impossible to touch upon the many other questions which the authors discuss, but it is safe to say that all readers of this book, whether they possess or control, or only work in a laboratory, will at least be brought to think about these problems, and cannot fail to be interested and stimulated by the clear and outspoken views set forth. CECIL H. CRIBB.

ALCOHOLIC FERMENTATION. By ARTHUR HARDEN, Ph.D., D.Sc., F.R.S. London: Longmans, Green & Co. **1923**. Price 6s. 6d. net.

The third edition of this work constitutes a most useful epitome of a subject of singular fascination alike to the investigator and the technologist. Before discussing Prof. Harden's contribution, the reviewer feels that he may be permitted to express the thanks of chemists to the author of the valuable series of Monographs on Biochemistry and to the publishers for issuing volumes at a reasonable price with a necessarily restricted sale. In these days of specialisation the sub-dividing of bio-chemistry into branches and the publishing of monographs thereon is applicable not only to other fields of our science, but also to the technical side.

Prof. Harden provides an interesting historical introduction which, however, as in the first edition (1911), concludes with the general effect of Buchner's work on researches associated with the mechanism of fermentation. In a future edition this chapter might with advantage be amplified, and some indication given of how the author's own researches, together with those of C. Neuberg, H. Euler and A. Fernbach, have added to the historical structure.

A good account is given of zymase and its properties, and of the manner in which the co-enzyme and phosphorus fits into the machinery of fermentation. The rôle played by the yeast enzyme, carboxylase, which the reader will remember decomposes certain hydroxy and keto acids, among them being pyruvic acid, is lucidly described, and from this the author passes on to an account of the chemical changes involved in fermentation and to a discussion of the different theories which have been enunciated in recent years.

The chapter on by-products of fermentation will be read with interest by those chemists who have to deal with the analysis of potable spirits. The bearing of Ehrlich's classical work on the alcoholic fermentation of amino acids is of great importance to the technologist, for, as this chemist points out, "the great variety of the bouquets of wine and aromas of brandy, cognac, arrack, rum, etc., may be very simply referred to the manifold variety of the proteins of the raw materials (grapes, corn, rice, sugar, cane, etc.) from which they are derived."

In concluding his remarks on the mechanism of fermentation the author writes that "Buchner's discovery of zymase has opened a chapter in the history of alcoholic fermentation which is yet far from being completed. In every direction fresh problems present themselves, and it cannot be doubted that, as in the past, the investigation of the action of the yeast-cell will still prove to be of fundamental importance for our knowledge of the mode in which chemical change is brought about by living organisms."

A very full bibliography, which also functions as a name index, is not the least useful feature of the volume. Biochemists are indeed indebted to Prof. Harden for writing this monograph. It supplies a want, for the literature on the subject of fermentation is bewildering even to those zymotechnologists who are investigators in a modest manner. JULIAN L. BAKER.

THE CHEMISTRY OF RUBBER. By B. D. W. LUFF, F.I.C. Pp. viii.+242. London: Ernest Benn, Ltd. 1923. Price 25s. net.

In his preface the author, who is the chief research chemist of the North British Rubber Co., Ltd., states that his aim has been to deal as fully as possible with the chemical principles involved in the cultivation, the collection and the manufacture of rubber; at the same time "consideration has been given to the concomitant changes in physical properties accompanying the process of vulcanisation."

During the past decade the study of the chemistry and physics of rubber and of the processes employed in the rubber industry has made rapid strides, and the literature of the subject has grown to such an extent that any work dealing more especially with modern principles and practice is to be welcomed, provided that the compilation has been carried out on sound lines. In the latter respect Mr. Luff may be regarded as having achieved success. His review of recent literature is distinctly good, and carries us almost up to the date of his preface—October, 1923.

The first seven Chapters (90 pages) of the book (if we except the opening historical review) deal with the sources, composition and properties of rubber latex and of crude rubber. Chapters VIII. to X. describe the vulcanisation process, the factors affecting vulcanisation and the properties of vulcanised rubber. Chapters XI. on "Compounding Ingredients" and XII. on "Accelerators," will probably, by specialists, be regarded as extremely useful, for it is around these materials and their application that a large proportion of recent research has developed. The penultimate section constitutes a brief sketch of the manufacturing process, and the final Chapter (XIV.) is headed "Methods of Analysis." To most analysts the latter will probably prove to be somewhat disappointing; to the expert, because Mr. Luff scarcely touches upon the important subject of the interpretation of the analytical and physical data obtained in the course of commercial work, and to the general analyst, because of its somewhat attenuated nature. The analysis of crude rubber is dealt with at rather disproportionate length, while the examination of vulcanised rubber, compounding ingredients and the like is treated very briefly. Notwithstanding such criticism of points of detail, Mr. Luff may be congratulated on having produced a work modern in its outlook and scientific in the handling; a book well worthy of taking its place in the library of all those interested in rubber. In conclusion, it may be stated that the printing and binding are exceptionally good, and that the 16 full plate illustrations are not only well chosen, but admirably reproduced.

PHILIP SCHIDROWITZ.

Publications Received.

QUANTITATIVE CHEMICAL ANALYSIS. By F. CLOWES & J. B. COLEMAN. 12th Edition. London: Churchill. 1924. Price 18s. net.

PHARMACEUTICAL AND FOOD ANALYSIS. By A. THURSTON. London: Chapman & Hall. 1923. Price 21s. net.

THE VEGETABLE PROTEINS. By T. B. OSBORNE. London: Longmans, Green & Co. 2nd Edition. 1924. Price 9s. net.

THE CHEMISTS' YEAR BOOK, 1924. Edited by F. W. ATACK. Manchester: Sherratt & Hughes. Price 21s.

APRIL, 1924. Vol. XLIX., No. 577.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held on Wednesday, March 5th, at the Chemical Society's Rooms, Burlington House. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. John Joseph Bryant, Edgar Wilfred Deag, Harold Wilton Hewis, B.Sc. (Lond.), A.I.C., Ernest Wilfrid Jackson, F.I.C., Thomas McGrath, and Alfred Scholes, F.I.C.

Certificates were read for the second time in favour of:—Messrs. Philip Walter Alloway, Lorentz Oliver Brekke, B.Sc. (Leeds), A.I.C., Alec Munro Cameron, B.Sc. (Edin.), F.I.C., Thomas William Drinkwater, L.R.C.P., L.R.C.S. (Edin.), F.I.C., John Ralph Furlong, Ph.D. (Wurzburg), A.I.C., Ernest Griffiths-Jones, M.Sc. (Manc.), A.I.C., Basil Gordon McLellan, F.I.C., and William Thomas Rigby, F.I.C.

The following were elected Members of the Society:—Messrs. Hugh Browning Brown, A.I.C., Sidney Augustus de Lacy, A.I.C., A.M.I.Ch.E., Joseph Henry Lane, F.I.C., B.Sc. (Lond.), Leslie Herbert Lampitt, D.Sc. (Birm.), F.I.C., Reginald Francis Moon, B.Sc. (Bristol), Maximilian Nierenstein, D.Sc. (Geneva), Ph.D. (Berne), William Simpson Shaw, M.Sc., A.I.C., Robert Norman Wright, A.R.C.S., B.Sc. (Lond.), A.I.C., Misses Phyllis Honor Price, B.Sc. (Bristol), and Mabel Susanne Lavinia Snelus, A.I.C.

The following papers were read:—"The Composition and Examination of Beef and Malt Wine," by G. D. Elsdon, B.Sc., F.I.C.; "Effect of Fatty Diet on the Composition of Butter Fat," by J. C. Drummond, D.Sc., F.I.C., and H. J. Channon, B.A., B.Sc.; "What is Bondon Cheese?" by G. D. Elsdon, B.Sc., F.I.C.; "Cream Cheese," by T. R. Hodgson, M.A., F.I.C., and "Some Facts on the Composition and Decomposition of Eggs," by R. T. Thomson, F.I.C., and J. Sorley, F.I.C.

Death of a Member.

We regret to record the death of Edgar Richards, Newport, R.I., on January 21, 1924.

Osmium Tetroxide as a Reagent for the Estimation of Tannins and Their Derivatives.

By C. AINSWORTH MITCHELL, M.A., F.I.C.

(*Read at the Meeting, February* 6, 1924.)

APART from its use as a stain for fat in microscopy, osmium tetroxide, or "osmic acid," as it is commonly termed, is but little used in analytical work, and, even its more common reactions are unknown to most chemists.

When making experiments as to the possibility of using it as a reagent for developing latent finger prints on paper (ANALYST, 1920, **45**, 124), I tried the effect of various photographic chemicals as possible intensifiers, and so accidentally discovered that pyrogallol combines with osmium tetroxide to form a compound which is reddish violet in dilute solution and almost black when concentrated, and that this compound can be used as an ink which does not have to undergo oxidation before forming a black stain on the paper.

The reaction is extremely sensitive, and is capable of detecting as little as 1 part of pyrogallol in 2 millions of water.

The intensity of the coloration is also proportional to the amount of pyrogallol present, and so, by the use of a technique similar to that which I devised for the estimation of pyrogallol, gallic acid and gallotannin by means of a ferrous tartrate reagent (ANALYST, 1923, **48**, 1), the reaction can be applied to the colorimetric estimation of pyrogallol.

In the ferrous tartrate method, the estimation is based upon the fact that the violet coloration produced by the tannin material apparently affords a measure of the pyrogallol groups, and that the reaction is not given by many compounds, such as phloroglucinol, in which the hydroxyl groups are in another position.

Catechol and catechol tannins give a dull olive green coloration in neutral solution, and catechol gives a violet coloration in slightly alkaline solution, with the ferrous tartrate reagent, and the possibility of using that reagent for the estimation of that group of tannins is now being studied by Miss Price under our Analytical Research Scheme.

Schluttig and Neumann (*Die Eisengallustinten*, 1891, p.) assert that only compounds with three adjacent hydroxyl groups combine with iron salts to form inks, *i.e.* compounds leaving a permanent stain on paper, but they themselves provide an exception to their rule when they state that catechol (a dihydroxy compound) yields an ink which is darker and more permanent than those produced by gallic acid or gallotannin.

Extending my experiments to other tannin materials, I have found that osmium tetroxide not only gives a violet coloration with compounds such as gallotannin, gallic acid and pyrogallol, with three adjacent hydroxyl groups in their molecule, but also with catechol, the exception overlooked by Schluttig and Neumann. It gives no coloration with phenol, salicylic acid, phloroglucinol, or resorcinol.

METHOD OF ESTIMATION.—The method I have used throughout is essentially the same as that previously described, the colours being compared in Nessler tubes provided with side tubulures and taps. The reagent consists of the ordinary 1 per cent. solution of "osmic acid" diluted with 10 parts of water.

The standard for the comparison is made by dissolving 0·1 grm. of pure pyrogallol, catechol or gallic acid in 100 c.c. of water. One c.c. of this solution is added to 100 c.c. of tap water, and treated with 1 c.c. of the dilute reagent, and the resulting coloration is matched, after the lapse of 5 minutes, with that given by 1 c.c. of a 0·1 per cent. solution of the unknown substance.

As in the case of the ferrous tartrate reagent, the nature of the coloured compound produced appears to vary with the proportion of the reagent, and subsequent dilution will not change one compound into another. It is advisable, therefore, that the coloration should keep below the limit of intensity of that formed by 1 c.c. of a 0·1 per cent. solution of gallic acid. It is also necessary that the solution should not be acid, but a slight excess of alkali does not affect the result. If tap water is used, the addition of any alkali is usually unnecessary. Otherwise, 1 c.c. of 0·1 *N* sodium carbonate solution may be added.

COMPARISON OF CATECHOL AND PYROGALLOL.—Osmium tetroxide, unlike the ferrous tartrate reagent, does not give different colorations with pyrogallol and catechol, but produces a similar violet coloration with each. The advantage of this is that it enables a simultaneous estimation to be made of the two compounds in the presence of each other, and the results can be expressed in terms of either.

Pyrogallol reacts much more rapidly than catechol, but the initial reactions appear to be complete in about 5 minutes. After about 15 minutes the colour begins to darken, apparently through absorption of oxygen.

For my original catechol standard I used a small, particularly pure specimen, made by Kahlbaum, which was kindly given to me by Dr. Nierenstein, and for the later experiments I used a supply of British origin. In the osmium tetroxide test 0·1 grm. of this catechol was colorimetrically equivalent to 0·098 grm. of the German preparation, and its degree of purity is therefore 98 per cent.

In a series of colorimetric comparisons I found that, as the average of a large number of readings, 87 c.c. of a 0·01 per cent. solution of pure pyrogallol was colorimetrically equivalent to 100 c.c. of a corresponding catechol solution.

The ratio between these values is as 1:1·14, and, if we compare the molecular weights of the two compounds, it will be seen that this is also the ratio between them.

		Mol. Weight.	Ratio.
Catechol	$C_6H_4(OH)_2$	110	1
Pyrogallol	$C_6H_3(OH)_3$	126	1·145

Hence, the entire substances appear to take part in the colour reaction, and the addition of one hydroxyl group to the catechol molecule increases its tinctogenic power by an amount corresponding to the increase in the molecular weight.

REDUCING ACTION OF CATECHOL AND PYROGALLOL.—On adding a few drops of strong hydrochloric acid to the coloured liquids obtained in the reaction, the pyrogallol solution turns bright blue, whereas the catechol solution becomes first olive green and then bright green. This is not due to any inherent difference between the two compounds, but to the fact that pyrogallol has a stronger reducing action than catechol on osmium tetroxide.

The bright blue coloration is due to the formation of osmous chloride, $OsCl_2$, whilst the green coloration is due to the formation of a mixture of this chloride with osmium tetrachloride, $OsCl_4$, which has a red colour. By increasing the amount of catechol in the solution, it, too, gives the bright blue coloration, which is also given by gallic acid and gallotannin.

CATECHOL AND PROTOCATECHUIC ACID.—A comparison of pure catechol with pure protocatechuic acid in 0·01 per cent. solution, for which I am also indebted to Dr. Nierenstein, gave the following results:

Protocatechuic Acid. c.c.		Catechol. c.c.
60	=	41
40	=	27

This gives a colorimetric ratio of 1:1·40 between the two compounds, which, again, is the relationship between the molecular weights:

Catechol.	Mol. Weight.	Protocatechuic Acid.	Mol. Weight.
$C_6H_4(OH)_2$	110	$C_6H_3(OH)_2.COOH$	154

$$\frac{154}{110} = 1{\cdot}40.$$

COMPARISON OF PYROGALLOL WITH GALLIC ACID.—The results obtained with catechol are, in themselves, an indication that the osmium tetroxide reaction is not concerned solely with the pyrogallic group, as seems to be the violet coloration given by the ferrous tartrate reagent with pyrogallol tannins. It was, however, somewhat of a surprise to find that gallic acid gave a distinctly darker coloration than pyrogallol—a result which indicates that the carboxyl group in the gallic acid also contributes to the reaction.

Pure crystalline gallic acid was used for the estimation, and the average results showed that 0·090 to 0·091 grm. of gallic acid was colorimetrically equivalent to 0·1 grm. of pyrogallol. The colorimetric ratio between the two substances is thus as 1:1·1.

Now, if we refer again to the respective molecular weights (pyrogallol, 126; crystalline gallic acid, 188), we find that the pyrogallic group in gallic acid ought to be responsible for 66·4 per cent. of the tinctogenic value and the carboxyl group for 24·1 per cent. (total 90·5 per cent.), whilst the water of crystallisation (9·5 per cent.) is inert.

The actual increase of tinctogenic power, however, on passing from pyrogallol to gallic acid was only 9·1 per cent., instead of 24·1 per cent., so that, in this case, there does not appear to be any exact numerical relationship between the tinctogenic values and the molecular weights of the two substances, and the factor 1·1 for converting pyrogallol results into terms of gallic acid must be accepted as an empirical one.

COMPARISON OF GALLIC ACID AND GALLOTANNIN.—The gallotannin which I used was that remarkable specimen in which no one has yet been able to detect more than 1 per cent. of glucose. The results obtained by me and by Mr. Ward have since been exhaustively confirmed by Dr. Nierenstein (ANALYST, 1923, **48**, 321; *Ber.*, 1923, 1876), and it is difficult to reconcile the existence of such a gallotannin with Fischer's theory of gallotannin as a galloyl glucose. I have shown (*loc. cit.*) that this gallotannin gives colorimetric results with the ferrous tartrate reagent in accordance with Nierenstein's formula for a gallotannin free from glucose, and this tannin was, therefore, a suitable one for comparison with gallic acid by the osmium tetroxide method. Here, again, I have found gallic acid to give a distinctly stronger reaction, the two compounds standing towards each other in the ratio of 1:1·1. It is not possible, as yet, to trace any definite relationship between these values and the formulæ.

CATECHOL AND CATECHINS.—Specimens of pure acacatechin, paullinia tannin and hemlock tannin given to me by Dr. Nierenstein, were compared with pure catechol. The ratio between the two first and catechol was in each case as 1:1·22, but hemlock tannin behaved very differently, and gave a ratio of 1:12·3. In view of our lack of knowledge of the exact constitution of these tannins, these values can only be regarded as empirical.

ESTIMATION OF GALLIC ACID IN PRESENCE OF GALLOTANNIN.—The method previously devised (*loc. cit.*) is also applicable in the case of osmium tetroxide. The two substances are first estimated colorimetrically together in terms of gallic acid, the tannin is then precipitated with quinine hydrochloride, and the gallic acid in the filtrate estimated as before.

By this means my standard gallic acid was found to contain 10·5 per cent. of gallic acid—a result which agrees with that previously obtained by the ferrous tartrate method. This procedure is also applicable to the estimation of other tannin derivatives not precipitated by quinine hydrochloride.

APPLICATIONS OF THE METHOD TO PYROGALLOL TANNINS.—I have proved that the method can be used for the estimation of tannin and gallic acid in various products containing pyrogallol tannins, such as galls, myrobalans, etc., but it offers no special advantages over the ferrous tartrate method in the case of these tannins. Some of the results which I have obtained with tea, however, are of special interest, since, in general, they agree with those obtained by the former method.

TANNIN SUBSTANCES IN TEA.—In each case 1 grm. of the tea was boiled with successive portions of water for 1½ hours, the united extracts made up to 100 c.c., and the tannin and gallic acid estimated as described.

The following results were obtained:

Tea.	Total Tannin Substances as Gallic Acid. Per cent.	Gallic Acid in Filtrate. Per cent.	Tannins in terms of Gallic Acid. Per cent.
China	6·0	1·14	4·86 × 1·1 = 5·34
Ceylon	11·1	2·0	9·1 × 1·1 = 10·0

So far, tea tannin does not appear to have been isolated in a pure condition, and we have, therefore, no definite factor for converting the results from their gallic acid to their tannin equivalent.

Smith (ANALYST, 1913, 38, 312) has shown that it is possible to obtain concordant results by the use of Chapman's method of precipitation with cinchonine sulphate, but he has not proved that his precipitate consists of pure cinchonine tannate. If tea tannin, like gallotannin, may be in some intimate association with glucose without that glucose forming an integral part of the molecule, it seems not improbable that the cinchonine precipitate contains not only the tannin, but also the associated glucose. In fact, Smith himself has shown that by dialysing the tannin separated from tea, a compound is obtained which combines with cinchonine in quite a different way from the original preparation.

CATECHOL TANNINS.—It is for the estimation of tannins in such substances as sawdust, coffee, and hops that the osmium tetroxide method fills a gap, for it enables either catechol tannins or mixtures of catechol and pyrogallol tannins to be estimated together in terms of catechol, pyrogallol, or gallic acid. These colorimetric values, once recorded, can afterwards be translated into their equivalents of the respective tannins as the constitution of these becomes known.

TANNIN IN SAWDUSTS.—One would hardly expect such a product as sawdust to be liable to adulteration, and yet, during the past year, I have had to examine several samples for a firm of bacon curers, to see whether they consisted of oak sawdust or contained deal, the resin in which would have been objectionable for the purpose required.

In addition to other tests, I made a series of estimations of the tannins in various sawdusts of known origin by the osmium tetroxide method, with the following results:

Sawdust.	Catechol Equivalent. Per cent.	Gallic Acid Equivalent. Per cent.	Tannin (Gallic Acid Equiv. × 1·1) Per cent.
Oak (English) I	9·5	7·9	8·69
,, ,, II	10·0	10·0	11·0
Oak (American) I	6·3	5·3	5·8
,, ,, II	4·2	3·5	3·8
Oak (Commercial) I	10·5	8·7	9·6
,, ,, II	—	9·0	9·9
Elm (English)	—	0·1	0·11
,, (Commercial)	0·7	0·58	0·63
Beech (English	—	0·01	0·01
Deal	—	0·005	0·005
Ash (English)	—	0·92	1·01
Mahogany (W. African)	—	10·6	11·6
,, (unknown)	—	3·2	3·52

It will be seen that the two commercial samples contained a proportion of tannin substances agreeing with that in genuine English oak sawdusts. In the case of the American sawdust, No. II., I estimated the amount of gallic acid, and found it to be less than 0·1 per cent.

An interesting point brought out by these estimations is the difference between American and English oak. It is well known that English oak is less suitable than American oak for making casks for beer or vinegar, and here we have the scientific explanation of the fact, namely, that the English wood contains much more tannin, and thus leads to turbidity if the beer or vinegar is put into casks which have only received the amount of seasoning which is sufficient for American oak.

Hops.—Through the kindness of Mr. J. L. Baker, I have been able to test the method upon typical samples of hops of known origin, and I find that it affords a very simple and rapid means of estimating the tannin substances present.

The following table gives some of the results obtained with 1 per cent. extracts of the hops:

	Hops.	Total Tannin Substances in terms of Gallic Acid. Per cent.	Factor.	Tannin. Per cent.
1.	Alsace	0·82	2·65	2·17
2.	St. Omer	1·42	,,	3·76
3.	Neve, Kent	(1) 0·96; (2) 1·00	,,	2·35
4.	Oregon	1·09	,,	2·88

Sample No. 3 contained 0·18 per cent. of tannin derivatives (expressed as protocatechuic acid) not precipitated by quinine hydrochloride.

Although hop tannin has not been isolated in a pure condition, and its constitution is still not definitely determined, it has been shown by Chapman (ANALYST, 1908, 33, 95; 1909, 34, 372) that the substance precipitated from an extract of hops by cinchonine sulphate is probably the pure tannin (less colouring matter), and so we have here a gravimetric standard for comparison. In the case of Sample No. 3, I estimated the tannin by Chapman's method, and obtained a cinchonine precipitate weighing 0·0428 grm., which, multiplied by Chapman's amended factor, 0·55, gives the amount of tannin as 2·35. These hops contained 0·89 per cent. of tannin giving a coloration with osmium tetroxide in terms of gallic acid, and, comparing this with the amount of tannin found by Chapman's gravimetric method, we obtain the factor 2·65 for converting the colorimetric results into lupulo-tannin.

Coffee.—Hitherto there has been no trustworthy method for estimating tannins in coffee, and even the cinchonine method has been found unsatisfactory in this case (Smith, ANALYST, 1913, 38, 316). The osmium tetroxide method, however, presents no difficulties, and I have estimated the tannin substances in a number of commercial products, including two samples of the same coffees, before and after roasting.

The following table gives results thus obtained:

Coffee.	Moisture. Per cent.	Total Tannins as Gallic Acid. Per cent.	Not precipitated by Quinine. Per cent.	Tannins in terms of Gallic Acid. Per cent.
Blend, Mysore and Costa Rica	—	(1) 3·47 (2) 3·57	—	—
Caffeineless Coffee	—	4·84	—	—
Costa Rica, raw	10·85	3·60	(1) 1·39 (2) 1·34	2·25
„ „ roasted	1·98	5·2	3·5	1·7
Nairobi, raw	—	3·56	1·68	1·86
„ roasted	—	3·60	2·45	1·15

It is interesting to note that a portion of the tannins is apparently decomposed during the roasting of the coffee and converted into derivatives which no longer give a precipitate with quinine hydrochloride.

CUTCH.—I have again to thank Dr. Nierenstein for letting me have small samples of genuine cutch typical of those sold in the Bombay market. A 1 per cent. extract of these gives a pronounced coloration with osmium tetroxide under the conditions described, but unfortunately the tint of the colour differs from that given by catechol, pyrogallol, or gallic acid, and so no exact comparison is possible in this case.

This drawback may be overcome when a pure specimen of catechu tannin has been prepared, but meanwhile all that is possible is to compare one sample of cutch with another, and so obtain a relative standard. I have done this in a number of cases, and have found that there is some relationship between the total colorimetric values of the different products. For example, taking one sample as 100, the colorimetric equivalent of another was 32.

The examination of cutch is also complicated by the difficulty of filtration, and by the fact that both the soluble and insoluble portions react with osmium tetroxide. Hence, until these difficulties have been removed, the colorimetric method cannot be recommended for the estimation of tannin in cutch.

DISCUSSION.

Mr. R. L. COLLETT pointed out that these colour reactions varied considerably with small changes in the hydrogen ion concentration, and asked whether the author had assured himself that his conditions in this respect were such as to give standard results.

Mr. A. CHASTON CHAPMAN said that he was interested in the relationship established between the colour reaction and his (the speaker's) cinchonine method, although he was becoming a little sceptical as to the value of colour reactions in general. He would like to know on what grounds the author regarded his standard tannin as exceptionally pure. Might it not be possible that glucose was, after all, an integral part of the tannin molecule, and that a substance free from glucose was not tannin at all.

Mr. C. A. **Mitchell**, in reply, said that he agreed that a control of the hydrogen ion concentration would add a degree of certainty to his method, although apparently even considerable variations in the amount of alkali added did not affect the colour. With regard to Mr. Chapman's question about the standard tannin: He had been informed by the manufacturers that it was a product of Chinese galls. It gave all the recognised tests for tannin, including the gold-beater's skin test and precipitation with quinine or cinchonine. Fischer had concluded that glucose was an integral part of the molecule, because, by his method of treatment, he had been unable to separate any more glucose from his preparations. But, as Trimble suggested many years ago, it seemed not improbable that tannin might be free from glucose, and that the presence of glucose in other preparations might be due to its being adsorbed, and not really in combination in the molecule.

The Plea for Standardisation.

By M. S. SALAMON, B.Sc.

(*Read at the Meeting, December* 5, 1923.)

During recent years the importance of commercial analysis has received increasing recognition, and as our knowledge of the composition of both organic and inorganic materials has been added to, so has there been an increasing tendency on the part of both the legislature and the commercial community to use such knowledge for the purpose of setting up legal standards of genuineness and commercial standards of purity and strength.

In the early days of commercial analysis, the number of methods available for quantitative determinations was strictly limited, the number of workers engaged on commercial analysis was few, and those that were so engaged were often pioneers in the subject, and thus, by force of circumstances, their decisions were but rarely challenged.

To-day, however, quite a large number of chemists are occupied in this particular field, and there are but few quantitative estimations in which the analyst has not a choice of a considerable number of processes for effecting the particular estimation that he has in view.

If chemical analysis were the exact science which many people, particularly laymen, believe it is, this multiplicity of methods would cause no trouble, but I think that it must be admitted that, in many cases, the particular method chosen for the analysis influences the result obtained, and analyses of the same material, but carried out by different methods, often show large discrepancies, and in many cases are not even comparable.

Even when only comparatively small discrepancies occur, they may be of considerable commercial importance, and are, moreover, frequently misunderstood and misinterpreted by the layman; with the result that trouble, inconvenience and expense are often caused, for which analysts in particular, and chemistry in general, get the blame.

In addition to the trouble that these differences may give rise to, there is that resulting from the lack of uniformity in expressing the results of analyses and the use of unqualified general terms, which have not received a precise and definite universal interpretation.

It is because of the recognition of these facts and the trouble, inconvenience and expense that they often cause, that attempts have been made to lay down precise details as to how the analysis of a particular material or materials is to be conducted, and precise definitions given so that the results may be expressed in terms which admit of no misinterpretation.

In this country this has only been done in very few cases, and my object to-night is to urge the wider and more general application of what, for want of a better word, is usually referred to as "Standardisation."

Owing to the non-adoption of standard methods the choice of the process to be used is left to the individual analyst, and I will give some examples to illustrate what this results in, and the difficulties that may arise therefrom.

I propose to consider first of all the question of the analysis of dried and condensed milk, and in this connection I would refer to the analyses given by Mr. J. H. Jephcott when reading his paper on "The Analysis of Dried Milk" (ANALYST, 1923, **48**, 529).

A sample of dried milk was divided into four parts and sent to different analysts; the results obtained were as follows:

	A.	*B.*	*C.*	*D.*
	Per cent.	Per cent.	Per cent.	Per cent.
Water	3·3	3·16	3·4	3·55
Ash	5·6	5·87	5·3	5·30
Fat	24·6	24·08	24·5	24·20
Lactose	40·6	40·67	38·6	43·30
Protein	25·6	25·36	25·9	23·65
Non-determined	0·3	0·86	2·3	Nil

All these analyses were carried out in laboratories which have had considerable experience of dried milk analysis.

As regards the results obtained by the analyst designated as *D*, I do not know very much about his methods, and I therefore do not propose to refer to his results in detail. As regards *A*, *B* and *C*, they are all known and recognised as careful workers, their estimations were made in duplicate, and every necessary precaution was taken in order to ensure the accuracy of their results.

Take the fat content, to start with, it will be noticed that *B* returns the fat content lower than either *A* or *C*; these latter are in quite fair agreement, and, as a matter of fact, used the same method, namely, the Werner-Schmidt; *B* used a different method, the Rose-Gottlieb.

This is by no means an isolated instance of the difference in the fat content which results from the use of different methods, and, as regards dried milk, I think that it is the invariable experience of all workers that the Rose-Gottlieb method gives a lower percentage of fat than does the Werner-Schmidt method.

It is true that the differences are not very large, but *B* gives a fat content practically half a per cent. lower than *A* and *C*, and such a difference may be quite sufficient to start a whole host of arbitrations and cause considerable expense, trouble and inconvenience to a number of dealers and manufacturers.

Moreover, a difference of this amount may become of even still greater importance in the near future, when the new regulations for dried milk come into force, and the description of "Full Cream Dried Milk" will acquire a definite legal and commercial meaning.

Now take the percentage of lactose; *A* and *B* both estimated this by optical rotation, and their results are in excellent agreement; the figure of 38·6 per cent. given by *C* was obtained by a gravimetric process, the solutions, method and table given in the Local Govt. Board Report on dried milk being used, but there appears a note on this analysis to the effect that if the lactose be estimated by the gravimetric American method and calculated on the American tables, its percentage comes out at 40·6 per cent., and the unestimated substance is correspondingly reduced to 0·3 per cent., this result being then almost identical with those of both *A* and *B*.

These particular estimations were repeated two or three times without any materially different results being obtained, and I have noticed similar differences between the two methods on other samples of dried milk.

There is the further question of what is meant when lactose is referred to in the analysis of dried and condensed milks. Does it mean hydrated lactose, anhydrous lactose or semi-hydrated lactose?

I have made enquiries of several well-known analysts who are perfectly familiar with these products (two of them being leading Public Analysts) and their opinions on the matter differ; some of them invariably calculate lactose to the hydrated form, others return it as anhydrous lactose, and another told me that as, in his opinion, it was by no means established, particularly in the case of dried milk, which form was present, he usually calculated it as semi-hydrated, although all of them agreed that they always described it as lactose without any qualification.

This lack of precise definition is not confined to these analysts, because, if you refer to the Local Govt. Board Report dealing with condensed milks, you will find there are pages of analyses given, but with no indication whatever as to which form of lactose is referred to, although in their report dealing with dried milks the lactose is specifically described as hydrated lactose.

In these circumstances what interpretation is to be put on the meaning of the words "milk solids" used in the regulations now in force for condensed milk?

There are other points in connection with the analysis of dried and condensed milks which merit attention, but I think that I have sufficiently indicated the need for standardisation in this branch, and I will pass on to consider quite another branch of analytical chemistry, namely, the commercial analysis of essential oils. Here, again, the use of different methods gives, even in the hands of the same analyst, different results, and slight modifications of the same method will give results that differ by as much as 2 per cent.

My own laboratory and that of Messrs. Crosfield have had occasion to investigate the estimation of so-called geraniol in Java citronella oil, and both of us have been astonished to find what different results are obtained by what are comparatively only slight modifications in detail.

For instance, a difference in weight of 0·2 grm. in the acetylated oil taken for saponification will sometimes mean a difference of over 1 per cent. in so-called total geraniol; the time during which the acetylated oil remains in contact with the dried sodium sulphate will also affect the final result.

Considering that commercial contracts are often made stipulating for a minimum percentage of so-called geraniol, and that allowances are claimed for any deficiency, it will be readily understood that a difference of 1 per cent. may be of considerable importance.

Mr. W. H. Simmons and Mr. C. T. Bennett have also called attention recently to the discrepancies that arise in connection with the estimation of phenols and aldehydes in essential oils, and the former has already put forward a strong plea for the standardisation of essential oil analyses.

In a joint paper by myself and Mr. Bennett, published a few years ago, we showed how the estimation of cineol in eucalyptus oil was affected by even slight modifications in details, and that different processes gave quite discordant results.

Yet you will find but few essential oil laboratories which agree on all the details of the methods employed.

Now it may be argued that, in the examples which I have given, I have chosen organic substances of complex composition, and that what holds good in their case does not necessarily apply to inorganic materials of somewhat simpler composition. That this argument does not hold is shown, I think, by the action in 1909, of what was then the Board of Agriculture, in issuing exact directions as to the methods to be used in analysing the different materials which came under the provisions of the Fertilisers and Feeding Stuffs Act, 1906, and the further issue, in 1918, of additional instructions for the same purpose.

Now the greater number of these methods refer entirely to the analysis of inorganic materials, many of which are of comparatively simple composition, and, in issuing these instructions, the Board evidently realised that, even with inorganic materials of simple composition, different methods might give different results.

The department in question not only apparently realised the importance of defining the method by which the six analytical results were to be obtained, but also published a list of permissible limits of error for the various estimations.

I could quote many further cases where discrepancies in analysis, due to the use of different methods, etc., occur, but as the majority of those present this evening can, doubtless, recollect many other instances within their own experience, I do not believe that it would serve any useful purpose for me to do so, particularly as the examples that I have given are of a sufficiently general character to show the confusion that exists at present, and to support my plea for wider and more general standardisation, which, I am convinced, is the only remedy.

It is, therefore, only a question as to what steps should be taken, and who should take them, in order that the existing state of affairs may be remedied, and it seems to me that this Society, representative, as it is to-day, of practically every branch of analytical chemistry, is particularly well fitted to take the initiative in this matter.

I do not suggest that we should at once attempt to standardise every analytical process, because I realise that it must take time, and can only be gradual, but I do think that we should make a beginning.

I would suggest, to start with, the formation of a small committee officially attached to this Society, which would act as a clearing house in the matter, and which might publish in a collected form those standard methods of analysis which have already been drawn up by different bodies. The committee might also endeavour to remedy obvious inconsistencies that exist at present as regards definitions; as, for example, the use of the word "oil" in oil-seed analysis, to denote petroleum spirit extract, and the use of the same word in oil-cake analysis to denote methylated ether extract.

Then steps might be taken to introduce standard methods for those articles which are in our immediate province, and which every day are forming the basis of commercial transactions.

In this connection I would particularly emphasise the desirability of the immediate standardisation of analytical methods for those articles for which the legislature has attempted to set up a legal standard of genuineness, and so give to those standards a real and definite meaning, and thus help to increase their usefulness.

Such a committee would be able to consider suggestions from analysts, manufacturers and the commercial community, in general, regarding analytical determinations, and thus act as a connecting link.

I am certain that the steps such a committee could take would reduce unnecessary disputes, make analyses more comparable, help to strengthen the enforcement of legal standards, and so render valuable service to the commercial community, the analytical profession and the State.

DISCUSSION.

The PRESIDENT, Mr. P. A. Ellis Richards, opened the discussion by reading the Council Minute of the 7th November on the subject of standardisation (*cf.* ANALYST, 1924, **123**).

Dr. BERNARD DYER said that there was the question of the limits of error, and referred to the Committee (of which he was a member) which was appointed to fix the official "limits of error," but this phrase did not refer essentially to errors of analysis, but rather to deviations from such causes as errors incidental to manufacturing, unavoidable lack of uniformity and sampling. For instance, in the case of certain feeding stuffs there was prescribed a limit of error of one-eighth of the quantity of albuminoids guaranteed, so that if the maker guaranteed

32 per cent. of albuminoids the guarantee would be fulfilled by a content varying from 28 to 36 per cent. That did not contemplate that two analysts working on duplicate samples might be expected to differ by 8 per cent. Although a satisfactory standard method for estimating anything might possibly be evolved, neither the person who worked the process, nor his skill in applying it could be standardised. He doubted the wisdom of adopting international standardisation.

Mr. John Myers said that, in his opinion, standardisation of nomenclature would be an excellent thing, and agreed that in certain cases it might be an advantage to report the method employed together with results obtained, but, on the whole, a general standardisation of processes would tend towards the elimination of initiative on the part of the chemist.

Dr. B. S. Evans said that he thought it desirable to differentiate between purely empirical processes and processes in which something definite had to be estimated; the thoroughly empirical Reichert process, for instance, should be standardised up to the hilt, but where some definite substance had to be estimated (as in the case of arsenic in food) the public wanted to know what was there, and it was no comfort to the authorities, the consumers, or the salesmen to know that it was estimated by a process which would give concordant results; they wanted to know the amount of arsenic actually there, and analysts must use the best method available.

Standardisation might be applied with advantage to certain of the physical determinations which analysts had to make. It was obviously impossible to use all the refinements of physical science, and the alternative was standard apparatus used in a standard way. Where this was done it should be indicated; for example, a viscosity determined in the Redwood manner might be called a "Redwood Viscosity."

If differences were really vital, as Mr. Salamon suggested they were becoming, there was this dilemma: that standardisation is proclaimed either as a counsel of perfection or as a means of "camouflaging" the analyst—and the latter is undesirable as it stops progress.

Mr. R. F. Innes said that the standardisation of methods of leather analysis and tannin analysis had been adopted some time ago, and had proved satisfactory, with certain modifications from time to time.

Mr. J. Allan said that the standardisation of tests might possibly lead to difficulties in certain directions, but it led to a great step in advance in processes. He referred to the Committee on Glycerin Analysis (which consisted of makers, users, and merchants of glycerin) and to the I.S.M. determinations. The methods adopted by that Committee were accepted as international standards, and their publication, in 1907, had completely stopped the disputes which had previously arisen between makers of glycerin, analysts, and the other people concerned. Of course methods of manufacture, which produced alterations in the crude material and altered in some way the general procedure, had to be taken into consideration; and changes had certainly taken place in the crude material since the Committee was first appointed, with the result that to-day the Committee was actually engaged in revising their methods. There was no reason, therefore, for standardisation causing methods to become stultified.

In his opinion standards set up by analysts for analysts were more desirable than the legal standards of a Government Department. He commended the step taken by the Society of endeavouring to exercise some supervision of processes; he thought they might go even further—many methods of analysis could very well do with standardisation.

Mr. E. R. Bolton said that he held no brief for or against standardisation —there was much to be said from either point of view. Given standardisation, there was always the difficulty of the people who did not carry out the standard processes after the manner described—with such one could not deal.

He felt that it was not practicable to expect a Committee to continue indefinitely carrying out investigations and re-standardising the processes for which standards were demanded. He thought the only practical way of dealing with the matter would be to ask such manufacturers and others, who had a special need for the standardisation of an analytical process, to call together a Committee of chemists to confer in the matter, and it was necessary that such chemists should be paid by the manufacturers or others who were interested in obtaining these standards for the regulation of their trade and for the saving of expense in arbitration and law disputes. Such Committees would put forward to a Committee of the Society the standard processes which their particular trade was prepared to accept, and these processes might be published under the auspices of the Society —the Society merely acting as a clearing house and organising body and taking no responsibility for the accuracy of the methods.

Mr. H. Jephcott said that standardisation would have very hearty support from the manufacturers. It was better, he thought, for the methods of analysis to be agreed upon by the analysts themselves than for the results of analyses to lead to actions in the Courts of Law—as would otherwise undoubtedly happen to an increasing extent, now that the food laws of the country were becoming more exacting.

Mr. E. T. Brewis said that, in his opinion, international standardisation would be very difficult; for instance, the methods of estimating alaloids were different in different countries.

Mr. W. H. Simmons referred to the British Engineering Standards Association who, he said, had found no difficulty in getting people to undertake the work of standardising processes and had arrived at satisfactory methods; their reports received the sanction of sundry Government Departments, and the standardised methods were constantly revised. In his opinion it was better that chemical methods should be standardised by analysts than by engineers.

Mr. Edward Hinks said he found himself agreeing with about two-thirds of the remarks of each speaker. There was quite a number of standard methods in use at the present time—more especially the British Pharmacopœia methods, which were very useful in many ways. When there was uncertainty it was useful to have a standard method, so that he was inclined to encourage their adoption. Regarding the danger, mentioned by another speaker, which came from incompetence—whatever method an incompetent worker used did not really affect the issue.

Mr. M. S. Salamon, replying, said there were other commercial people besides manufacturers who might be willing to pay for the standardisation of methods of analysis.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE SOLUBILITY OF MILK POWDER.

THE published methods for the determination of the solubility of milk powders are not satisfactory, for a number of reasons. For example, both Hunziker's method (*Condensed Milk and Milk Powder*, p. 321) and the method used at the Government Laboratory (vide *Food Reports*, 24, p. 171) fail, by reason of the difficulty of obtaining a representative sample when the powder has been taken into solution, owing to the separation, or partial separation, of fat. A further disadvantage in the Government Laboratory method is that it fails to differentiate between two powders which may show the same solubility at 60° C., but which at 20° C. would give very different solubilities.

In comparing the solubilities of two powders it is desirable to make due allowance for the moisture content and fat content of the two samples and to express the results as percentage solubility of the solids-not-fat.

The following method depends upon the estimation of the insoluble portion of the powder, and applies to both full cream and skim milk powders. Both full cream and skim milk powders should show a solubility of 99·8 per cent. or thereabouts. A milk powder of poor solubility will show a result as low as 63 per cent. :

METHOD.—(1) To about 38 c.c. of distilled water at 20° C. in a flask of 250 c.c. capacity add 5 grms. of full cream milk powder, or to 45 c.c. of water add 5 grms. of skim milk powder; cork and shake steadily for three minutes. Transfer the whole contents to a tared centrifuge tube, and whirl at about 1500 revolutions for three minutes. If full cream powder has been taken, now remove any layer of cream on the surface of the milk in the tube, and wipe any cream off the inside of the tube.

(2) Taking care not to disturb the deposit in the tube, pipette off about 5 c.c. of the liquid into a tared nickel dish of weight (w); weigh rapidly; say (a) = weight of dish plus fluid; dry for four or five hours in the steam oven, or about 5 minutes on the Mojonnier hot plate, and then for 15 minutes in the Mojonnier vacuum oven; weigh: say (b) = weight of dish plus milk solids.

$(a-b)$ = weight of water lost.
$(b-w)$ = weight of solids.

(3) Now decant as much as possible of the fluid without disturbing the residue, wipe off any cream, etc., on side of tube, and weigh the tube plus residue plus the small quantity of associated fluid: say weight of contents = (c). Then wash out the residue by means of a wash bottle into another tared dish. To hasten drying alcohol may be used to wash out the deposit. If the amount of insoluble matter is small, it may be dried in the tube instead of being washed out.

Dry in oven as in (2) and weigh.
Say (d) = weight of solids.
Then $(c-d)$ = weight of water lost.

CALCULATIONS.—Assume butter fat = [illegible] moisture = Z per cent.

Dissolved solids in the fluid contained in $($[illegible] $\times \frac{b-w}{a-b} = f$.

Therefore, weight of insoluble solids contained in $(c) = d - f = s$.

Whence, insoluble matter per cent. of powder $= 20s$,

and solubility of the powder $= (100 - 20s)$, or,

Insoluble matter per cent. of solids-not-fat $= \frac{20s \times 100}{100 - (Y+Z)}$,

and solubility of solids-not-fat $= 100 - \frac{20s \times 100}{100 - (Y+Z)}$.

EXAMPLE.—Five grms. full cream powder used.

Dish + 5 c.c. fluid (a) = 37·702 grms.
Dish + dry solids (b) = 33·176 grms.
Weight of water lost $(a-b)$ = 4·526 grms.

Dish + dry solids (b) = 33·176 grms.
Dish (w) = 32·604 grms.
Dry solids $(b-w)$ = 0·572 grms.

Centrifuge tube + holder + wet residue = 27·192 grms.
Centrifuge tube + holder + dry residue = 27·127 grms.
Weight of water lost = 0·065 grms. = $(c-d)$.

Centrifuge tube + holder + dry solids = 27·127 grms.
Centrifuge tube + holder = 27·114 grms.
Weight of dry solids in tube = 0·013 grms. = d.

Dissolved solids in the fluid in residue after centrifuging

$$= \frac{0{\cdot}065 \times 0{\cdot}572}{4{\cdot}526} = 0{\cdot}008 \text{ grms.} = f.$$

Weight of insoluble solids from 5 grms. of powder $= d - f$.
$= 0{\cdot}013 - 0{\cdot}008 = 0{\cdot}005 = s$.

Whence, solubility $(100 - 20s) = 99{\cdot}90$ per cent.

If powder contained 2 per cent. of moisture and 28 per cent. of fat, insoluble matter per cent. of solids-not-fat $= \frac{20 \times 0{\cdot}005 \times 100}{70} = 0{\cdot}143$.

And solubility of solids-not-fat $= 100 - 0{\cdot}143 = 99{\cdot}86$ per cent.

L. H. LAMPITT.
E. B. HUGHES.

CHEMICAL DEPARTMENT,
J. LYONS & CO., LTD., CADBY HALL,
KENSINGTON, W.14.

PUBLIC HEALTH (CONDENSED MILK) REGULATIONS, 1923.

TABLES FOR TESTING "EQUIVALENT PINTS" DECLARATION.

THE tables below may be found of service in testing the "equivalent pints" declaration on the labels of tins of condensed milk.

The method of operation is as follows:—The percentage of Total Milk Solids, Fat, or Milk Solids-not-fat in the condensed milk, as the case may be, is found in its appropriate left-hand column. Level with this figure, in a horizontal direction, will be found the minimum weights of condensed milk which will satisfy the declarations at the top of their respective columns. The weights are given in ounces and drams avoir. and grms., in each case to the next highest whole dram or grm.

An example will make the procedure clear. Let us assume a condensed milk (full-cream, sweetened) containing 31·0 per cent. of Total Milk Solids, and let the contents of the tin weigh 13 oz. 10 dr. The percentage of T.M.S. is found in the left-hand column of Table I. (the table for full-cream milks). Following the 31·0 line horizontally, we find that 13 oz. 10 dr. lies between the minimum weight for 1⅝ pints (13 oz. 7 dr.), and that for 1¾ pints (14 oz. 8 dr.). The label on the tin should therefore declare 1⅝ pints, since the weight found is not sufficient for 1¾ pints, and the Regulations do not recognise fractions smaller than ⅛ pint. A similar procedure is used when the calculation is to be made from fat or milk solids-not-fat.

In the case of full cream milks (Table I.), it has been pointed out by Hinks (ANALYST, 1923, **48**, 596) that the declaration on the label should be satisfied by calculations from both the fat and the total milk solids, otherwise milk of the prescribed standard has not been used in manufacture. Where no analysis has been made, the T.M.S. is taken, for the purposes of calculation, to be the minimum laid down by the Regulations (31·0 per cent.), and this line has been printed in italics for ease of reference. In calculating the table the sp. gr. of the Standard Milk (12·4 per cent. T.S., 3·6 per cent. fat) has been taken as 1031·8 (Richmond's formula). The columns for T.M.S. and fat can be treated as independent of one another for the purpose of calculation.

In the case of skimmed milks (Table II. for sweetened, Table III. for unsweetened), the calculation should be made from Milk Solids-not-fat (see Hinks, *loc. cit.*). When, however, no analysis has been made, the calculation has to be made from Total Milk Solids. For this purpose it has been assumed that the skimmed milk specified in the Regulations (containing not less than 9·0 per cent. of solids-not-fat) will contain 0·1 per cent. of fat, giving a minimum T.M.S. of 9·1 per cent. and sp. gr. (Richmond's formula) of 1035·4.

Calculating from this, it is found that skimmed condensed milk of the minimum T.M.S. laid down in the Regulations (26·0 per cent. for sweetened and 20·0 per cent. for unsweetened) will contain Milk Solids-not-fat of 25·71 per cent. and 19·77 per cent. respectively (*i.e.* a fat content of 0·29 per cent. and 0·23 per cent. respectively, figures which agree well with the author's experience). These lines also have been printed in italics.

For percentages, or declarations, not included in the tables, the minimum weights may be found accurately enough by simple proportion; *e.g.* a declaration of ¾ pint will require one-half the minimum weights given for a declaration of 1½ pints, the result in each case being rounded off to the next highest whole dram or grm.

It should be noted that the Regulations consider two distinct offences, one regarding the composition of the condensed milk, and one regarding the declaration of equivalent pints. If the weight found has to be searched for in the tables *above* the line in italics, at least one offence (that referring to composition) *must* have been committed (except in certain cases of skimmed milk where the fat is greater than 0·29 per cent. [Table II.] and 0·23 per cent. [Table III.]), and the other *may* have occurred. If, on the other hand, the weight has to be searched for *below* the line in italics, the offence as regards composition has *not* occurred (except in those cases of skimmed milk where the fat is less than 0·29 per cent. [Table II.] and 0·23 per cent. [Table III.]), whereas the offence as regards declaration *may* have occurred.

TABLE I.—FOR FULL CREAM MILKS, SWEETENED OR UNSWEETENED.

Analysis of Condensed Milk.		Minimum Net Weight of Condensed Milk.															
T.M.S.	Fat.	1⅛		1¼		1⅜		1½		1⅝		1¾		1⅞		2	
Per cent.	Per cent.	oz. dr.	grms	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.
30·0	8·71	9.10	273	10.11	303	11.12	333	12.13	363	13.14	393	14.15	424	16.0	454	17.1	484
·2	·77	9.9	271	10.10	301	11.11	331	12.12	361	13.13	391	14.14	421	15.15	451	17.0	481
·4	·83	9.8	269	10.9	299	11.10	329	12.11	358	13.11	388	14.12	418	15.13	448	16.14	478
·6	·88	9.7	267	10.8	297	11.8	326	12.9	356	13.10	386	14.11	415	15.11	445	16.12	475
·8	·94	9.6	265	10.7	295	11.7	324	12.8	354	13.9	383	14.9	413	15.10	442	16.10	472
31·0	*9·00*	*9.5*	*264*	*10.6*	*293*	*11.6*	*322*	*12.7*	*351*	*13.7*	*381*	*14.8*	*410*	*15.8*	*439*	*16.9*	*468*
·2	·06	9.4	262	10.4	291	11.5	320	12.5	349	13.6	378	14.6	407	15.7	436	16.7	465
·4	·12	9.3	260	10.3	289	11.4	318	12.4	347	13.4	376	14.5	405	15.5	434	16.5	462
·6	·17	9.2	259	10.2	287	11.3	316	12.3	345	13.3	373	14.3	402	15.3	431	16.4	460
·8	·23	9.1	257	10.1	286	11.2	314	12.2	342	13.2	371	14.2	400	15.2	428	16.2	457
32·0	·29	9.0	256	10.0	284	11.0	312	12.0	341	13.0	369	14.0	397	15.0	426	16.0	454
·2	·35	9.0	254	9.15	282	10.15	310	11.15	338	12.15	367	13.15	395	14.15	423	15.15	451
·4	·41	8.15	252	9.14	280	10.14	308	11.14	336	12.14	364	13.14	392	14.13	420	15.13	448
·6	·46	8.14	251	9.13	279	10.13	306	11.13	334	12.13	362	13.12	390	14.12	418	15.12	446
·8	·52	8.13	249	9.13	277	10.12	305	11.12	332	12.11	360	13.11	388	14.10	415	15.10	443
33·0	·58	8.12	248	9.12	275	10.11	303	11.11	330	12.10	358	13.10	385	14.9	413	15.9	440

TABLE II.—FOR SKIMMED SWEETENED MILKS.

Milk Solids-not-Fat in Condensed Milk.	Minimum Net Weight of Condensed Milk.															
	1⅜		1½		1⅝		1¾		1⅞		2		2⅛		2¼	
Per cent.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.
24·4	10.9	298	11.8	325	12.7	352	13.6	379	14.6	407	15.5	434	16.4	461	17.4	488
·6	10.7	296	11.6	323	12.6	350	13.5	376	14.4	403	15.3	430	16.2	457	17.1	484
·8	10.6	293	11.5	320	12.4	347	13.3	373	14.2	400	15.1	427	16.0	453	16.15	480
25·0	10.5	291	11.3	318	12.2	344	13.1	370	14.0	397	14.15	423	15.14	450	16.13	476
·2	10.3	289	11.2	315	12.1	341	13.0	367	13.14	394	14.13	420	15.12	446	16.11	472
·4	10.2	287	11.1	313	11.15	339	12.14	365	13.13	391	14.11	417	15.10	443	16.9	469
·6	10.1	284	10.15	310	11.14	336	12.12	362	13.11	388	14.9	413	15.8	439	16.7	465
25·71	*10.0*	*283*	*10.14*	*309*	*11.13*	*334*	*12.11*	*360*	*13.10*	*386*	*14.8*	*412*	*15.7*	*437*	*16.5*	*463*
·8	9.15	282	10.14	308	11.12	333	12.11	359	13.9	385	14.8	410	15.6	436	16.5	461
26·0	9.14	280	10.13	305	11.11	331	12.9	356	13.8	382	14.6	407	15.4	432	16.3	458
·2	9.13	278	10.11	303	11.9	328	12.8	353	13.6	379	14.4	404	15.2	429	16.1	454
·4	9.12	276	10.10	301	11.8	326	12.6	351	13.4	376	14.2	401	15.1	426	15.15	451
·6	9.11	274	10.9	298	11.7	323	12.4	348	13.3	373	14.1	398	14.15	423	15.13	447
·8	9.10	272	10.7	296	11.5	321	12.3	346	13.1	370	13.15	395	14.13	419	15.11	444
27·0	9.8	270	10.6	294	11.4	319	12.2	343	13.0	367	13.13	392	14.11	416	15.9	441

TABLE III.—FOR SKIMMED UNSWEETENED MILKS.

Milk Solids -not-Fat in Condensed Milk. Per cent.	Minimum Net Weight of Condensed Milk.															
	1		1⅛		1¼		1⅜		1½		1⅝		1¾		1⅞	
	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.	oz. dr.	grms.
18·4	10.3	288	11.7	324	12.11	359	13.15	395	15.4	431	16.8	467	17.12	503	19.0	539
·6	10.1	285	11.5	320	12.9	356	13.13	391	15.1	427	16.5	462	17.9	498	18.13	533
·8	9.15	282	11.3	317	12.7	352	13.11	387	14.14	422	16.2	457	17.6	492	18.10	527
19·0	9.13	279	11.1	313	12.5	348	13.8	383	14.12	418	16.0	452	17.3	487	18.7	522
·2	9.12	276	10.15	310	12.3	345	13.6	379	14.9	413	15.13	448	17.0	482	18.4	516
·4	9.10	273	10.13	307	12.1	341	13.4	375	14.7	409	15.10	443	16.14	477	18.1	511
·6	9.9	270	10.12	304	11.15	337	13.2	371	14.5	405	15.8	439	16.11	472	17.14	506
19·77	*9.7*	*268*	*10.10*	*301*	*11.13*	*334*	*13.0*	*368*	*14.3*	*401*	*15.6*	*434*	*16.8*	*468*	*17.11*	*501*
·8	9.7	267	10.10	301	11.13	334	13.0	367	14.2	401	15.5	434	16.8	468	17.11	501
20·0	9.5	265	10.8	298	11.11	331	12.14	364	14.0	397	15.3	430	16.5	463	17.8	496
·2	9.4	262	10.7	295	11.9	327	12.12	360	13.14	393	15.0	426	16.3	458	17.5	491
·4	9.3	260	10.5	292	11.7	324	12.10	357	13.12	389	14.14	421	16.0	454	17.3	486
·6	9.1	257	10.3	289	11.5	321	12.8	353	13.10	385	14.12	417	15.14	449	17.0	481
·8	9.0	255	10.2	286	11.4	318	12.6	350	13.8	382	14.9	413	15.11	445	16.13	477
21·0	8.15	252	10.0	284	11.2	315	12.4	346	13.6	378	14.7	409	15.9	441	16.11	472

These tables have been calculated for use in the laboratories of Messrs. Lipton, Ltd., and the author wishes to express his thanks to Mr. J. W. Black, chief chemist, for permission to publish them, and for suggestions regarding the form in which they have been drawn up.

R. E. Essery.

Analytical Department, Messrs. Lipton, Ltd.,
Rouel Road, S.E.16.

THE REICHERT-MEISSL VALUE OF ALMOND OIL AND APRICOT KERNEL OIL.

Ross and Race (Analyst, 1911, **36**, 263) give the Reichert-Meissl value of almond oil and apricot kernel oil as 2·6. As these figures are very different from those found by Hawley and one of us (*Year Book Pharm.*, 1913, 573), and as it seemed difficult to understand such a high figure from oils having a composition such as these oils have, a number of further determinations have been made.

Sixteen samples of almond oil, obtained from various sources, have been examined, and the highest Reichert-Meissl value obtained was 0·2. In the case of eleven of the samples the figure was 0·1, whilst in four of the samples it was 0·0.

Two samples of apricot kernel oil had a Reichert-Meissl value of 0·1 in each case, whilst two samples of peach kernel oil gave 0·1 and 0·0 respectively.

The Polenske values of these oils were determined in each case, with results as shown in the following table:

Polenske Value.	Number of Samples. Almond.	Apricot Kernel.	Peach Kernel.
0·2	2	–	–
0·3	5	1	1
0·4	4	1	–
0·5	2	–	1
0·6	1	–	–
0·7	1	–	–
1·0	1	–	–

It is a little difficult to understand the high figure obtained by Ross and Race, although, as they do not give the acidity of their samples, it might conceivably be due to rancidity, as it is well-known that rancid oils give a high Reichert-Meissl value; but such oils are usually so rancid that they would not be accepted, even superficially, as normal samples. Another possible explanation is that the glycerin used in saponification contained a proportion of volatile acids, as a number of years ago it was not generally recognised that some samples of glycerin gave blanks as high as 3·0. This suggestion is supported to a certain extent by the fact that the almond oil and apricot kernel oil gave the same figure.

Whatever be the true explanation, it would appear fairly certain that these figures are abnormal, and do not represent those given by ordinary oils; in fact, it is not usual for fresh seed oils, with the exception of castor oil, to give Reichert-Meissl values of much more than about 0·5.

G. D. ELSDON.
PERCY SMITH.

MUNICIPAL LABORATORY,
SALFORD.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE FOURTH QUARTER, 1923.

THE total number of samples examined during the quarter was 1245, of which 1069 were analysed under the Sale of Food and Drugs Acts. Of these, 844 were bought informally, of which 16 were adulterated; 225 samples were bought under the provisions of the Acts, and of these 3 were adulterated.

MILK.—Of the 585 samples examined, 10 contained less than 11·5 per cent. of total solids.

CONDENSED MILK.—Six samples were taken for analysis under the new Regulations. Of these, 5 were properly labelled, but one was marked "Skimmed" on the declaration, and "Machine-skimmed" on another part of the label. As it only contained 0·4 per cent. of milk fat the declaration was incorrect, as *un-condensed* skimmed milk may contain about 1 per cent. of fat. The vendor was cautioned.

The Regulations appear to be defective in making no distinction between "skimmed" and "machine-skimmed" products. In fact, one is not aware of any condensed skimmed milk being on the English market.

EGG SUBSTITUTE POWDER.—Five samples were, as usual, little more than coloured baking powders. In one case tartaric acid was used, and in the others acid phosphate. Two samples, claiming to be "complete substitutes" for eggs, contained only 0·2 and 0·3 per cent. of fat respectively. The vendors were cautioned.

Borax.—Six of seven informal samples contained from 1 to 3 parts of arsenic per million, but the seventh contained 100 parts per million, and ought to have been labelled as unsuitable for internal use. The vendor was cautioned.

An aluminium pan, which had been stained by boiling water in it, was examined for the Water Department. It was found that water from the works in Wales did not stain aluminium, but that harder waters from local streams or deep wells did produce a stain.

J. F. Liverseege.

COUNTY OF KENT.

Report of the Agricultural Analyst for the Fourth Quarter, 1923.

During the quarter 400 samples of fertilisers and 33 samples of feeding stuffs were submitted for analysis. Of the former, 138, and of the latter, 10 were found unsatisfactory.

Shoddies.—The total number examined was 320; of 177 samples sold with a guarantee, deficiencies of ammonia were found in 110 samples. This unsatisfactory state of affairs could be remedied if the guarantee given at the factory could be attached to the sample taken at the farm, but at present the Fertilisers and Feeding Stuffs Act only concerns the last seller, and a vendor is exonerated from liability to prosecution if he purchases a fertiliser with a written warranty which contains a false statement subsequently used, provided that he has no reason to believe the statement to be false and that the fertiliser is sold in the same state as he received it

Fur Waste.—A sample, guaranteed to contain 12 per cent. of ammonia, contained only 6·46 per cent.; it was also unsatisfactory owing to the fur being in very large pieces and containing waste cotton wool.

Feather Wastes.—One of the samples, sold as feather dust, contained nearly 50 per cent. of mineral matter or dirt, whereby its ammonia content was reduced to 8·7 per cent.

Flue Dust.—A sample, sold at a relatively cheap rate, contained 12·6 per cent. of potash (=23·3 per cent. of potassium sulphate). Washed with water the sample yielded 14·8 per cent. of potassium sulphate, and the remainder of the potash was easily soluble in dilute acids. Field trials with flue dusts have shown that apparently they have a higher manurial value than that due merely to soluble potash.

Decorticated Earthnut Cake.—A sample was low in albuminoids, owing to the fact that no shell had been removed from the nuts, so that the cake was really undecorticated.

The presence of as little as 0·1 per cent. of castor seed in some earthnut cake caused havoc in a dairy herd.

F. W. F. Arnaud.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

CHESHIRE CHEESE.

On January 14th, R. D. Johnson was summoned at Ashton-under-Lyne for selling Cheshire cheese not of the nature, substance and quality demanded. The Inspector gave evidence that he had seen on the defendant's stall at Ashton market a large cheese labelled "Finest Cheshire Cheese," and that he had bought a pound of this cheese for 1s. 2d. under the provisions of the Act.

The official certificate of the Analyst embodied the following analysis and opinion:—"Water, 54·9; fat, 14·0; mineral matter, 4·3; proteids (casein, etc.), 24·4; and lactose, etc., 2·4 per cent. This is not a sample of Cheshire cheese, but is a cheese made from part-skimmed milk. This opinion is based upon the fact that genuine Cheshire cheese contains, at least, 24 per cent. of fat, whereas the sample contains only 14 per cent. of fat. Alternatively, genuine Cheshire cheese contains, when expressed on the water-free sample, at least 45 per cent. of fat, whereas the above sample, similarly expressed, contains only 31 per cent. of fat."

The defendant said that what was known to the trade as Cheshire cheese was cheese made under a particular system in Cheshire or adjoining counties. On his stall was Dutch cheese at 1s. 2d. and Cheshire cheese at 1s. 6d., and his assistant might not have heard the inspector ask for Cheshire cheese.

The magistrates imposed a fine of £5, with 2½ guineas costs.

SHREDDED SUET.

On February 12, Messrs. Pearks, Ltd., were summoned at the Tower Bridge Police Court for the sale of shredded suet, not of the nature, substance and quality demanded.

The packet purchased by the inspector was labelled "Britox Shredded Suet."

Mr. Ryall, for the prosecution, stated that the mixture was not declared as a mixture at the time of purchase, and, on analysis, was found to contain 76·7 per cent. of fat, 20·2 per cent. of rice flour, and 3·1 per cent. of moisture. This showed 0·6 per cent. excess of moisture, and 2·7 per cent. excess of rice flour over the mixture allowed by the now obsolete "Shredded Suet Order." He contended that there could be no warranty of a mixture such as the label indicated, and that, the Order having been annulled, it was for the Court to decide. The statement on the invoice that the article was "A mixture of suet and rice flour" could not be a warranty of the contents of the packet.

The Magistrate (Mr. Fry) said that the sale was admitted, and there was no denial that there was an admixture of rice flour in the packet. No expert evidence had been called to assist the Court, and even the prosecution did not suggest that the rice flour and moisture were much in excess. The case would be dismissed.

Dominion Laboratory, New Zealand.

FIFTY-SIXTH ANNUAL REPORT OF THE DOMINION ANALYST FOR THE YEAR 1922.

THE report of Dr. J. C. Maclaurin, of the work done in the Dominion Laboratory during the year 1922, gives particulars of the 4579 samples analysed for different Government departments, including 2637 for the Public Health Department, 597 for the Customs, 789 for the Mines Department, and 150 for the Explosives Branch.

JUSTICE.—Samples from the Justice Department comprised liquor, drugs, brandy chocolate and exhibits relating to cases of suspected poisoning. The amount of brandy in the chocolates was approximately 0·5 per cent. Poisons found in three exhibits were, respectively, arsenious oxide, cresylic acid, and potassium cyanide, each in sufficient quantity to cause death.

DEPARTMENT OF HEALTH.—Foodstuffs in great variety and some medical preparations were submitted for analysis. Preservatives were found in some foods in which they are not permitted. Boric acid was detected in four samples of bacon and three of whitebait, and salicylic acid in a lemon squash.

Samples of borax were found to contain from 10 to 350 parts of arsenic per million.

Flour and Oatmeal.—Samples of oatmeal and wheat meal preparations were analysed to enable comparisons of their nutritive values to be made. The following results were obtained:

OATMEAL AND OAT PREPARATIONS.

N/159 (1–7) were oatmeal and oat preparations. No. 1, oatmeal: a very coarse meal consisting of large uniform particles. No. 2, oatmeal: fairly finely ground meal, but coarse particles of husk present. No. 3, porridge-meal: a finely ground meal with a few particles present. No. 4, prepared meal: finely ground meal with fewer coarse particles than No. 3. No. 5, prepared meal: in thin concave flakes, roughly circular in shape; diameter, ⅛–3/16 in. No. 6, prepared meal: in large flakes; the cleaned grain flattened; average size, ⅜ by ¼ in. No. 7, rolled oats: similar in appearance to No. 6.

Analyses.

Results expressed as Percentages.

	(1.)	(2.)	(3.)	(4.)	(5.)	(6.)	(7.)
Water lost at 100° C. ..	8·35	8·97	8·71	8·40	9·10	8·78	9·95
Fat	7·45	7·16	8·33	7·77	8·54	9·05	7·14
Ash	1·48	1·73	1·63	1·43	1·40	1·73	1·60
Phosphoric anhydride ..	0·83	0·92	0·82	0·80	0·82	0·90	0·87
Nitrogen	1·96	1·85	2·10	2·03	1·89	2·31	2·03
Protein (N × 6·31) ..	12·37	11·67	13·25	12·81	11·93	14·57	12·81
Crude fibre	1·10	1·60	1·70	1·40	1·00	1·30	0·90
Hot water soluble, 35° C.	5·03	6·93	6·73	4·50	4·70	6·95	3·90
Cold water soluble, 10° C.	4·20	4·60	4·53	3·68	4·45	4·43	3·45
Difference	0·83	2·33	2·20	0·87	0·25	1·52	0·45
Starch (by difference) ..	69·25	68·87	66·38	68·19	68·03	64·57	67·60

The analyses show that in none of these preparations has the process of milling materially reduced the percentage of fat, or of phosphoric anhydride. In milling wheat the phosphoric anhydride is reduced from 1 per cent. in whole wheat to 0·20 per cent. in patent flour.

If the vitamin content runs parallel to the phosphoric anhydride (as it does in wheat and corn products) the above oat products are all equally rich in vitamins, and not inferior in that respect to the unmilled oats.

The very small differences in the water soluble at 10° C. and 35° C. show that in no case has any great alteration been made in the starch by heating. This was confirmed by microscopic examination.

WHEATMEAL AND FLOUR.

N/1988, 2197, 2294 (1, 2), 2295 (1, 2), 2823 (1, 2), 2824 (1, 2), 2825 (1, 2): Wheatmeal and flour. For convenience these were renumbered 1 to 12 in consecutive order.

Analyses.

Results expressed as Percentages.

	(1.) Wheat-meal.	(2.) Wheat-meal.	(3.) Wheat-meal.	(4.) Wheat-meal.	(5.) Flour.	(6.) Flour.
Water	13·90	13·44	13·18	12·97	13·75	13·46
Fat	1·69	1·98	1·95	1·87	1·14	0·94
Protein ($N \times 6{\cdot}25$)	10·50	9·00	8·50	9·40	8·40	10·10
Ash	1·53	1·69	1·19	1·55	0·49	0·52
Phosphoric anhydride	0·740	0·970	0·587	0·689	0·268	0·287

	(7.) Wheat-meal.	(8.) Flour.	(9.) Flour.	(10.) Wheat-meal.	(11.) Wheat-meal.	(12.) Flour.
Water	13·92	13·58	13·38	13·40	13·40	13·60
Fat	1·82	1·00	0·96	1·78	1·65	1·03
Protein ($N \times 6{\cdot}25$)	10·00	9·60	9·00	10·00	10·50	9·00
Ash	1·85	0·54	0·55	1·77	1·50	0·46
Phosphoric anhydride	0·880	0·287	0·236	0·829	0·740	0·287

It will be seen that the amount of fat in the whole meals is almost twice that found in the flours. The reduction in the amount of mineral matter is, however, more striking, the flours containing only one-third of the amount of mineral matter present in the wheatmeals. The phosphoric anhydride has suffered a corresponding reduction. If it is established that the vitamin content of flour runs parallel to the phosphoric anhydride, the above results show that the white flours sold in New Zealand are very deficient in vitamins when compared with the whole meals. It should be noted that the lowest proportion of phosphoric anhydride (0·236 per cent. in No. 9) is higher than the lowest (0·20 per cent. in Virginia patent flour) found by Voegthin and Meyers in American flour. It is probable that No. 3 is not entirely whole meal.

CHLOROFORM.—Ten samples were examined, and all complied with the B.P. tests except one, which gave a distinct reddish colour when shaken with sulphuric acid. This was in a corked bottle, and it is suggested that unprotected corks should not be allowed in containers for anæsthetic chloroform, and that the manufacturers should be required to put the date of manufacture on the bottles.

Ether for Anæsthesia.—Nine samples were examined. None complied with the B.P. test for acidity, and one contained a very large amount of free acid, and gave marked reactions for peroxides. In the case of two samples of the same make the test for peroxides showed none in the newer preparation, but traces in the older one. This emphasises the necessity of discarding old preparations for anæsthetic use. As suggested in the case of chloroform, the date of manufacture ought to be stated on the bottle.

Institute of Chemistry of Great Britain and Ireland.

FORTY-SIXTH ANNUAL GENERAL MEETING: 3RD MARCH, 1924.

At the 46th Annual General Meeting of the Institute of Chemistry, held at Russell Square to-day, the Meldola Medal, the gift of the Maccabaeans, was presented to Mr. C. N. Hinshelwood, B.A. (Oxon). The Medal is awarded for the work of most promise published by a British chemist under thirty years of age, brought to the notice of the adjudicators during the year.

Mr. A. Chaston Chapman, F.R.S., the retiring President, in his address, referred to the growing activity of the Institute during his three years of office. The roll of membership had increased by 1129, and about 1000 new chemists had been absorbed into useful professional life. He emphasised the point that the Institute endeavoured to counteract the modern tendency to turn out narrow and imperfectly educated specialists. He deplored the tendency on the part of Government departments to undervalue professional scientific and technical service, especially in view of the fact that the public chemical service is becoming every year a more important part of the machinery of government.

Dealing with the proposals for closer co-operation amongst chemical societies, which he thought should have the warm support of all, he expressed the hope that in any scheme of co-operation the Institute would not sink any of its individuality. The general public was coming more and more to recognise in chemistry one of the most powerful factors in the creation of material wealth, at a time when it is more important to create wealth than to quarrel about the distribution of what little the war had left us. He quoted Mr. Baldwin, the late Prime Minister, who, in a recent speech at Glasgow, had said that under the stimulus of the war we had made great headway in pure chemistry, and we had schools of chemistry in this country which compared with any in the world. We should take care that our industries absorbed the output of those schools, and should not be content to run only rule-of-thumb industries and leave those more highly organised industries, which depend upon science and brain power, to the foreigner. He felt that those words had a very special significance with reference to the reported negotiations between the British Dyestuffs Corporation and the Interessen Gemeinschaft.

The new President, Prof. G. G. Henderson, F.R.S., Regius Professor of Chemistry in the University of Glasgow, was formally installed.

The Officers, Council and Censors for 1924–25 were elected.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Composition of "Tasajo." A. Bickel and J. A. Collazo. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 360–363.)—"Tasajo" is a preserved meat exported from Rio de la Plata, and consists of salted and pressed flesh which has been sun-dried. A similar unsalted prepartion consumed by the natives, but unsuitable for export, is known as "charque." The following analyses of tasajo are given:

	Per Cent.	Per Cent.
Water	20·98	20·90
Albuminoids	23·25	20·48
Fat	45·15	45·00
Ash	13·51	—
(including salt)	11·63	—

Biological experiments proved that "tasajo" is rich in vitamins *A* and *B*.

H. E. C.

Estimation of Rye Flour in Wheat Flour. J. König and F. Bartschat. (*Zeitsch. Unters. Nahr. Genussm.*, 1923, **46**, 321–339.)—The authors have thoroughly investigated chemical methods for the estimation of these two flours when present in admixture, and show that the diastase method proposed by several workers (*e.g.* Amberger, ANALYST, 1922, **47**, 73), methods dependent on the gluten content, on the solubility of the non-glutenous protein, or on the protein soluble in 20 to 70 per cent. alchohol are not reliable for the analysis of the mixed flours. It is shown, however, that the percentage of the total protein soluble in saturated solution of calcium sulphate is constant for each flour, and independent of the actual amount of protein in any particular sample of the flours. Ten grms. of the flour are moistened in a 500 c.c. flask with the saturated calcium sulphate solution at the room temperature (0·22 grm. in 100 c.c.), made up to the mark and shaken in a machine for one hour, after which the nitrogen is estimated in 100 c.c. of the clear filtrate and expressed as percentage of the total nitrogen in the flour. Treated in this way, wheat flour shows 29·1 per cent., and rye flour 51·5 per cent., so that the proportion of the two flours may be interpolated from the following table:

Wheat.	Rye.	Per Cent.	Wheat.	Rye.	Per Cent.	Wheat.	Rye.	Per Cent.
100	—	29·1	60	40	38·06	20	80	47·02
90	10	31·34	50	50	40·30	10	90	49·26
80	20	33·58	40	60	42·54	—	100	51·50
70	30	35·82	30	70	44·78			

The average experimental error with the method is about 5 per cent., but may be exceeded in the case of flour of unusually high acidity.

H. E. C.

Rapid Analysis of Sugars. Purification and Concentration of Enzyme Solutions. F. W. Reynolds. (*J. Ind. Eng. Chem.*, 1924, **16**, 169–172.)—Yeast extracts may be decolorised and clarified by dialysing the crude extracts in collodion dialysing sacks for twenty-four hours, or longer, against running water; 4 drops of acetic acid per 100 c.c. of extract are then added and, after fifteen hours, the flocculent precipitate produced is removed by filtering the extract through paper. The clarified extract thus obtained is concentrated by filtering off the required amount of water through a collodion membrane filter; the enzyme is retained on the filter and thereby obtained in concentrated form. It is quite practicable, in this way, to prepare invertase solutions, which, when used in the proportion of 10 c.c. per 100 c.c. of sucrose solution, will hydrolyse the sucrose completely within fifteen minutes at ordinary temperature. W. P. S.

Compounds developed in Rancid Fats. W. C. Powick. (*J. Agric. Res.*, 1923, **26**, 323–362.)—The Kreis test with phloroglucinol and hydrochloric acid is the chemical reaction which gives results most nearly parallel with organoleptic tests for rancidity; but application of the test to a large number of substances which have been suggested as the cause of rancidity shows that the fatty acids, their corresponding aldehydes, hydroxy-stearic acids, acrolein, methyl-glyoxal, dihydroxyacetone and oleic acid ozonide, do not give the reaction and are not the cause of the phenomenon. Oxidation of oleic acid produces a typical rancidity; hence the glyceryl radical is not essential. A number of substances give reddish or pink colours with phloroglucinol and hydrochloric acid as, for example, cotton seed oil, but such colours are distinguishable spectroscopically from the colour of true rancidity; this latter has a maximum extinction coefficient in hydrochloric acid solution at a wave length of approximately 5400; hence, if the colour obtained with the Kreis test is examined spectroscopically, the reaction becomes a true criterion. Epihydrin-aldehyde, an unstable compound formed by the interaction of acrolein and hydrogen peroxide which has not been completely separated, gives the colour of rancidity and is its probable cause, although nonyl aldehyde is responsible for the characteristic smell. It is suggested that the epihydrin compound is formed by the atmospheric oxidation of oleic acid, with the concurrent formation of heptyl and other aldehydes which have been isolated from rancid fats. A review of previous work, together with a bibliography, is given. H. E. C.

Oil of *Cyperus esculentus*. J. Pieraerts. (*Matières Grasses*, 1924, **16**, 6674–6681.)—A detailed study of the oil pressed out from the fleshy tubercles at the end of the rhizome of this plant (found in Mediterranean countries) is given. They contain 20 to 27 per cent. of a liquid pale yellow edible oil having the following characteristics:—Sp. gr. at 15°, 0·9176; solidifying point, liquid at 1° C.; saponification value, 191·3; unsaponifiable matter, 0·62 per cent.; iodine value, 76·9; Reichert-Meissl value, 0·2; free fatty acids as oleic, 0·85 per cent.; rotatory power, +0·06. The low temperature at which stearine is deposited, together with the good keeping qualities, enhances the value of the oil for edible purposes.

The oil is non-drying, and the lead salt ether separation shows approximately 80 per cent. of liquid and 20 per cent. of solid fatty acids. The liquid acids had an iodine value of 84·6 and consisted chiefly of oleic acid, and the solid acids (approximately 60 per cent. of palmitic and 40 per cent. of myristic acids) had an iodine value of 75·1. For further detailed analysis the original paper should be consulted. It also includes references to the distribution of the genus *Cyperus* in the Congo.

W. F. Baughman and G. S. Jamieson (*J. Agric. Res.*, 1923, **84**, 77–82, and *J. Soc. Chem. Ind.*, 1924, **43**, B139) agree, in the main, with the characteristics of the oil given above, but they found the oil (obtained by extraction with petroleum spirit) to consist of the glycerides of the following acids: Oleic, 73·3; palmitic, 11·8; stearic, 5·2; linolic, 5·9; arachidic, 0·5; lignoceric, 0·3 per cent.; and myristic, a trace. D. G. H.

Vernine in Green Leaves and Berries of the Coffee Plant. T. de A. Camargo. (*J. Biol. Chem.*, 1924, **58**, 831–834.)—The berries of the coffee tree in varying stages of development, while yet green, contain caffeine, adenine, hypoxanthine, xanthine and vernine (guanosine), this last in relatively large quantities. Experimental details are given of work carried out on the green leaves and berries of the coffee tree of the species "arabica." In these exists a pentoside containing guanine. This is probably the guanosine discovered in several plants by Schulze, and by Levene and Jacobs in nucleic acid. This pentoside is probably the origin of the caffeine in the green leaves and berries of the coffee tree. The guanosine is transformed to guanine, then to xanthine, and, lastly, from xanthine to caffeine, by the action of enzymes. P. H. P.

Estimation of Paraffin Oil in Pharmaceutical Preparations. J. Weichherz and Z. Klinger. (*Chem. Zeit.*, 1924, **4**, 20.)—Paraffin oil emulsions are generally prepared by means of carbohydrates or albumins. Comparative results are given showing the percentages of oil, as estimated by extraction in a Soxhlet apparatus for a minimum time of 36 hours, and also by the following method:—Five grms. of the emulsion are treated with 100 c.c. of a 35 per cent. potassium hydroxide solution and kept gently boiling for half an hour. The solution is cooled and shaken with 50 c.c. of petroleum spirit, which separates completely in one or two minutes, after which 25 c.c. are pipetted off, evaporated in a tared flask, and the residue heated to constant weight at 105° C. This rapid method is not free from error, which, however, lies between the limits of 0·66 and 1·45 per cent. The preparations examined included "Cristolax," "Laxamel," "Semprolin," and two emulsified by means of milk with malt extract and gum arabic. Hydrolysis of the emulsifying agent by means of strong sulphuric or hydrochloric acids was not satisfactory, as this treatment caused much darkening in colour, and the shaking with the petroleum spirit caused a stable emulsion. Different samples of the above trade products showed much variation in the paraffin oil content, *e.g.* "Cristolax," 44 to 51 per cent.; "Semprolin," 77 to 80·5 per cent.; and "Laxamel," 59 to 63 per cent. R. F. I.

Biochemical, Bacteriological etc.

Absorption of Metallic Salts by Fish. A. Thomas. (*J. Biol. Chem.*, 1924, 58, 671–674.)—Experiments with nickel salts were carried out on a number of fish of the species *Fundulus heteroclitus*. The preparation of the fish for analysis is described, and also the method of analysis. Fish were kept in sea water to which nickel chloride had been added, and a table gives the amount of nickel absorbed from solutions of different concentrations in a given time by *Funduli*, in terms of metallic nickel in the dried material, as found by analysis. *Funduli* absorb a considerable proportion of nickel from solutions of the chloride in sea water without evidence of poisoning. They exhibited no signs of sickness, even after 2 weeks in solutions of $N/250$ concentration. In fresh water, however, nickel chloride is toxic, even though the fish are accustomed to fresh water. A solution of $N/8{,}000$ concentration caused death in a few hours. Nickel was eliminated from *Funduli* after the fish had been kept in running sea water for 8 days, subsequent to their immersion in a $N/500$ solution of nickel chloride in sea water for 90 hours. P. H. P.

Estimation of the Hydrogen Cyanide Content of Amygdalin. J. H. Roe. (*J. Biol. Chem.*, 1924, 58, 667–669.)—The author's method for estimating simple, soluble cyanides (*J. Amer. Chem. Soc.*, 1923, 45, 1878) is applied to the estimation of the hydrogen cyanide produced from amygdalin by enzymic hydrolysis and application of the aeration procedure, and this method should prove a favourable means for quantitative investigations of cyanogenetic plants in general. The procedure is as follows:—An apparatus suitable for aeration is prepared. In one flask a 0·10 grm. sample of amygdalin is placed, about 0·05 grm. of the enzyme emulsin added, and then 100 c.c. of water, and a few drops of amyl or capryl alcohol. A stopper is now inserted, the aeration tubes are closed by means of rubber tubing and pinch-cocks, and the flask is thoroughly shaken and warmed to 45° for 15 minutes. This flask is then connected with a second flask containing 100 to 150 c.c. of 5 per cent. sodium hydroxide, and attached to a suction pump. A current of air is passed through the flask containing the amygdalin mixture into the one containing alkali, at the rate of about 3 litres per minute, thus carrying the freed hydrogen cyanide over, aeration is continued for about 3 hours, and then the flasks are disconnected. After 10 drops of 10 per cent. potassium iodide have been added to the alkaline cyanide solution in the second flask, as an indicator, it is titrated with 0·01 N silver nitrate until a faint turbidity appears. The reading of the burette, multiplied by 0·0005404, gives the number of grms. of hydrogen cyanide yielded by the sample of amygdalin. It is important to notice that the enzyme continues to work while aeration is in progress. P. H. P.

Destruction of Vitamin *B* by Age. G. M. Findlay. (*Biochem. J.*, 1923, 17, 887–890.)—Samples of Indian lentils and peas kept for 38 years still contained appreciable quantities of vitamin *B*. In contradistinction to the observations of

Ghose (*Biochem. J.*, 1922, 16, 35), however, they appeared to have lost a small amount of their vitamin *B* content, even if allowance was made for the slight differences of technique in the experiments described (*e.g.* the rats fed with the lentils were somewhat lighter in weight than those used by Ghose). The author could not obtain fresh lentils for comparison with the old seeds. Graphs of the results of his experiments are given. Seeds which have lost the power of germination have not necessarily lost all their vitamin *B* content. The whole question of the destruction of vitamin *B* with age is obviously one which requires further and fuller investigation, more especially in regard to the effects of different climatic conditions, *e.g.* heat and moisture, on the rate of destruction. P. H. P.

Alleged Colour Reaction for Vitamin *C*. H. D. Kay and S. S. Zilva. (*Biochem. J.*, 1923, 17, 872–874.)—Recently Bezssonoff (ANALYST, 1921, 46, 462) devised a reagent—a modification of Folin's reagent for phenol—which he claimed gave a specific colour reaction for the antiscorbutic factor, because with certain antiscorbutically active substances it gave a positive reaction. The authors have employed this reaction on a great many substances which have been simultaneously tested on guinea-pigs, and have decided that the test is not reliable enough to be of any use in the detection of the vitamin. They describe experiments showing that an antiscorbutically active substance, *viz.* decitrated lemon juice (adsorbed with "norit") fails to give the colour reaction, and certain antiscorbutically inactive substances, *viz.* yeast and yeast-extract, produce the blue coloration in question. Similarly, when the blue coloration obtained with an active substance is compensated in a comparator with untreated marmite, the same olive-green coloration is produced as that obtained with the reagent and a yeast preparation, marmite. It is obvious that the reagent is not specific for the detection of vitamin *C*. It is of interest to notice that the substance which gives the blue reaction is, like the vitamin, destroyed by oxidation. P. H. P.

Some Chemical Reactions of the Substance Containing Insulin. H. A. Shonle and J. H. Waldo. (*J. Biol. Chem.*, 1924, 58, 731–736.)—The pancreatic substance containing insulin used in this work was prepared from the pancreas of hogs. Attempts were made to isolate the physiologically active substance as a compound possessing a definite chemical constitution, or to discover some characteristic reaction peculiar to the active principle. Neither of these results was achieved, but disconnected data were secured. From these the authors conclude that the pancreatic substance containing insulin appears to be a complex mixture of proteoses, which give typical protein reactions. Numerous unsuccessful attempts were made to reactivate insulin (destroyed by a brief warming in an alkaline medium) by warming with acid, and also to reactivate reduced insulin by oxidation. It was also found that precipitation methods are inadequate to purify insulin to the point of securing a substance of constant composition. Further research must determine whether the active principle is a proteose, or is merely intimately associated with a proteose fraction. P. H. P.

Active Chlorine as a Germicide for Milk. H. Hale and W. L. Bleecker. (*J. Agric. Res.*, 1923, **26**, 375–381.)—The germicidal activity in milk of chlorine from chlorine water, and from sodium and calcium hypochlorites has been compared. The results show that chlorine water is much the most efficient; one part of chlorine in 1000 reduced the total bacteria in milk from 1,500,000 to 7000 per c.c., and killed all *B. coli* in 45 minutes, and 1 in 3000 reduced the bacterial content to 160,000; at this concentration the flavour is only just perceptible. Chlorine from sodium or calcium hypochlorite only attains this efficiency in 1½ and 19 hours, respectively, and both salts seriously affect the flavour. The germicidal action is not strictly proportional to the concentration. The *o*-toluidine test for free chlorine is not applicable to milk, and gives no colour with quite high concentrations; the starch-iodide reaction has a limit of sensitiveness in milk of about 1 in 160,000. H. E. C.

Toxicological and Forensic.

Contamination of Beverages and other Food with Zinc. J. W. Sale and C. H. Badger. (*J. Ind. Eng. Chem.*, 1924, **16**, 164.)—Analyses of bottled "root" beer, which had produced vomiting immediately after it had been consumed, showed that the beverage contained 229 mgrms. of zinc per litre. The metal had possibly been dissolved from galvanised pails in which the beer had been kept before being bottled. Other cases of zinc poisoning due to the consumption of foods which had been in contact with galvanised vessels are recorded, and the authors have determined the rate at which zinc is dissolved by various liquids from ordinary galvanised iron pails; the results obtained are given in the following table:

	Zinc mgrms. per litre.		Acidity c.c. $N/10$ acid per litre.	
	After 17 hrs.	After 41 hrs.	After 17 hrs.	After 41 hrs.
Tap water	5	21	0	0
Distilled water	9	27	1	0
Carbonated water	193	181	348	96
Milk	438	1054	—	1109
Orangeade	530	854	397	533
Lemonade	1411	2700	493	366

The increase in the acidity of the orangeade was due to the formation of carbon dioxide by fermentation. W. P. S.

Agricultural Analysis.

The Perchlorate Method for the Estimation of Potassium in Soils. H. J. Page. (*J. Agric. Sci.*, 1924, **14**, 132–138.)—High results in the estimation of potash in soils and fertilisers when using the perchlorate method led to the examination of commercial perchloric acid, and it was found that when chloric acid is present, even to a comparatively small extent, high results are obtained, due to the partial insolubility of barium and potassium chlorates in alcohol. The perchloric acid method is quite as accurate as the well-known platinum method, but it is essential to test the acid for chlorate, which is readily done by boiling the diluted acid with ferrous sulphate and testing with silver nitrate. It is also shown that when estimating potash in soil deficient in lime it is sufficient to add 0·1 grm. of calcium carbonate, instead of 0·5 grm. as recommended by Neubauer. This effects a considerable saving of perchloric acid. H. E. C.

Estimation of Nitrates in Soils by the Phenoldisulphonic Acid Method. H. J. Harper. (*J. Ind. Eng. Chem.*, 1924, **16**, 180–183.)—In the method described clear and colourless soil extracts, free from organic substances (which yield a coloration with the reagent) are obtained by treating the extract with copper sulphate and calcium hydroxide. Fifty grms. of the soil are shaken for ten minutes with 250 c.c. of water, containing 5 c.c. of $N/1$ copper sulphate solution; 0·4 grm. of calcium hydroxide and 1 grm. of magnesium carbonate are then added, the mixture is shaken for five minutes, and filtered. Ten c.c. of the filtrate (or more if the nitrate content is very small) are evaporated to dryness in a basin, the residue, when cold, is treated with 2 c.c. of phenoldisulphonic acid, and the basin is rotated so that the reagent comes into contact with all the residue. After ten minutes 15 c.c. of water are added, the solution is cooled, rendered slightly ammoniacal, and the coloration is compared with that produced by a known amount of nitrate. If the soil contains more than 15 parts of chloride per million, the 250 c.c. of water with which the extract is made should also contain 10 c.c., or more, of 0·4 per cent. silver sulphate solution. The phenoldisulphonic acid is prepared by dissolving 25 grms. of pure phenol in 150 c.c. of concentrated sulphuric acid, adding 75 c.c. of fuming sulphuric acid, and heating the mixture at 100° C. for two hours. W. P. S.

Estimation of Tannin in Plant Tissues. P. Menaul. (*J. Agric. Res.*, 1923, **26**, 257–258.)—Twenty grms. of the ground grains or tissue are thoroughly extracted with petroleum spirit for at least 12 hours, and 200 c.c. of 95 per cent. alcohol are added to the dry residue, and allowed to stand for 16 hours, with occasional shaking, and then filtered. To 10 c.c. of the filtrate 2 c.c. of 10 per cent. lead acetate solution are added, the mixture is warmed to 75° C., and then centrifuged, and the liquor decanted. The tannin in the residue is dissolved, and the lead precipitated by the addition of 5 to 10 drops of 5 per cent. sulphuric acid. The mixture is now diluted and centrifuged, and the clear solution diluted to 50 or 100 c.c. To this is added 2 c.c. of the reagent described below, and the intensity of colour is matched against standard solutions containing 1 or 2 mgrms. of tannin in 50 c.c. The reagent is prepared by boiling 100 grms. of sodium tungstate, 30 grms. of arsenious oxide, 300 c.c. of water, and 50 c.c. of hydrochloric acid under a reflux condenser for 2 hours, and then diluting to 1 litre. The colour is affected by reducing agents, but not by phenols, sugars or proteins, and is stable for about 1 hour; it is not specific for tannins, but these are the only substances producing a colour after the above process of extraction. H. E. C.

Organic Analysis.

Evaluation of Purity of Various Organic Products by the Dichromate Method. E. C. Grey. (*Biochem. J.*, 1923, **17**, 768–771.)—The author discusses his own dichromate method (*J. Chem. Soc.*, 1914, **105**, 2204) for the volumetric estimation of carbon and that of Martin (*Rev. Intern. Falsif.*, 1904, **17**, 48; *J. Chem. Soc.*, *Abstr.*, ii, 520) for the estimation of alcohol. The former measures the carbon dioxide produced and, when necessary, the acetic acid formed, whilst the latter measures the dichromate reduced, and is more convenient to employ. Provided the oxidation products are known, the dichromate method could be used for estimating many substances. By combining the two methods, two constants can be obtained, which permit of the estimation of the proportions of substances. In calculating the dichromate value of any aliphatic substance, the rule is that the substance is completely oxidised to carbon dioxide, except the carbon atom of a methyl group and the next adjacent carbon atom, which appear as acetic acid. Results of experiments described, measuring the dichromate reduced, support the writer's theory that substances devoid of methyl groups should yield no acetic acid, but only carbon dioxide and water. Succinic acid is apparently the one exception to the rule. The oxygen equivalent of any substance can, therefore, be calculated and tables constructed for mixtures of any two substances. The proportions of a mixture of two alcohols can be ascertained, and also the proportions of two acids in a mixture, provided there is a difference in their oxygen value. P. H. P.

Iodimetric Estimation of Osazones. D. R. Nanji. (*Biochem, J.*, 1923, **17**, 761–763.)—During the study of the decomposition of osazones with hydrochloric acid it was observed that so long as the ratio of the nitrogen to the hydrochloric acid used was fixed, the percentage of the osazone decomposed was constant. By keeping the amount of hydrochloric acid constant and varying the amount of nitrogen present, a curve was obtained which gave very concordant results when applied to different sugar osazones. This is the basis of the following empirical method for the estimation of nitrogen in sugar osazones: The phenylhydrazine formed is estimated by the iodimetric method of Ling and Nanji (*Biochem. J.*, 1921, **15**, 466). From 5 to 60 mgrms. of the osazone are treated in a Freudrich flask with 10 c.c. of *N* hydrochloric acid, the mixture is heated in a boiling water bath for 1 hour, then washed into a beaker, and to it 10 c.c. of *N* sodium hydroxide are added. It is next acidified with a drop of dilute acetic acid, and the solution made alkaline with an excess of pure sodium bicarbonate solution. The alkaline solution is then washed into an Erlenmeyer flask containing an excess of a known volume of standard 0·02*N* iodine solution. The excess of the iodine is found by titration with a standard solution of sodium thiosulphate. A table is given showing the amounts of nitrogen which correspond to different numbers of c.c. of 0·02 *N* iodine solution used. P. H. P.

Essential Oil of Manuka. *(Leptospermum scoparium.)* R. Gardner. (*J. Soc. Chem. Ind.*, 1924, **43**, 34–35T.) This shrub is very plentiful in New Zealand; the oil distilled from the leaves and branchlets is pale greenish-yellow in colour. It showed $[n]_D$, 1·50; Sp. gr. at 15° C., 0·921; with a range of b. pt. from 160°–270° C. The approximate composition of the oil was found to be phenols (leptospermol), 2·8; terpenes, 2·8; esters of cinnamic acid, (calculated as ethyl cinnamate), 4·8; other esters (acetic etc., esters of alcohol, unidentified, of rose odour) calculated as $CH_3COOC_{10}H_{15}$, 12·9; semi-solid, non-volatile matter, 7·7; sesquiterpene (by difference), 69·0 per cent. The sesquiterpene gives similar reactions to aromadendrene, but yields a definite liquid monohydrochloride.

D. G. H.

Rutin from the Flowers of Elder. C. E. Sando and J. U. Lloyd. (*J. Biol. Chem.*, 1924, **58**, 737–745.)—Details are given of the preparation and purification of a yellow pigment from the white flowers of the elder (*Sambucus canadensis* L.) which was briefly described by Lloyd (*Eclectic Med. J.*, 1920, **80**, 591) and tentatively called "eldrin." The authors have attempted to establish its exact identity and to determine its composition. Figures show the resemblance between crystals of eldrin and crystals of *Eschscholtzia* rutin. Results show that purified eldrin has the same empirical composition as rutin; that upon hydrolysis it yields quercetin, glucose and rhamnose; that the quantity of quercetin obtained, with a slight difference shown by investigation to be caused by the presence of an impurity, difficult to eliminate, agrees with the quantity theoretically expected from rutin; and that spectroscopically eldrin agrees with authentic rutin from *Eschscholtzia*. The authors feel justified therefore, in concluding that eldrin, the yellow pigment from the flowers of *Sambucus canadensis* L., is rutin. ($C_{27}H_{30}O_{16}$.) P. H. P.

Application of a new Reaction of Resorcinol to the Detection of Nitroprussides and Ammonia. M. Caseneuve. (*Bull. Soc. Pharm. Bordeaux*, 1923, No. 3; *Ann. Chim. anal.*, 1924, **6**, 43–44.)—If the reagent of Legal (nitroprusside, sodium hydroxide and acetic acid) is applied to a solution containing at least 1 per cent. of resorcinol, a green coloration results. Substitution of sodium by ammonium hydroxide renders the reaction more delicate. According to the quantities of reagents used, the presence of resorcinol, nitroprusside and ammonium hydroxide may be detected. *Resorcinol*:—To 2 or 3 c.c. of a solution containing 10 c.c. of a recently prepared 10 per cent. solution of sodium nitroprusside, 5 c.c. of a saturated solution of sodium acetate and 10 c.c. of ammonium hydroxide, are added a few drops of resorcinol solution. A green or blue-green coloration develops with as little as 0·5 mgrm. of resorcinol, and the reaction is not produced by other phenols. *Nitroprusside*:—To 2 or 3 c.c. of a 10 per cent. solution of resorcinol in ammonium hydroxide (scarcely yellow if the resorcinol is pure) are added a few drops of a solution of a nitroprusside or a particle of the solid salt. The presence of 0·1 mgrm. of nitroprusside gives rise to a blue-green coloration, which also results from salts of zinc, cadmium, nickel and cobalt, but, in the latter cases, the colour is not altered by adding an excess of sodium hydroxide, which, in the case of the nitroprusside, causes a change to yellow. *Ammonia gas and volatile amines.* A drop of a freshly made solution of 2 grms. of sodium nitroprusside, 1 grm. of resorcinol, and enough water to dissolve them, held on the end of a glass rod, turns blue or blue-green in the presence of these gases. In the case of ammonium salts the ammonia is first liberated by means of sodium or potassium hydroxide. D. G. H.

Estimation of Cellulose in Wood by the Chlorination Method. G. J. Ritter and L. C. Fleck. (*J. Ind. Eng. Chem.*, 1924, **16**, 147–148.)—Two chlorination methods were investigated, the long method (20, 15, 15, 10 and 10 minutes' chlorination) and the short method (5, 5, 5, and 5 minutes' chlorination). The removal of lignin from the cellulose was about the same in both methods, indicating that the reaction between lignin and chlorine is fairly rapid; the substance, "lignin chloride," soluble in sodium sulphite solution, must be removed from the surface of the wood particles, since it is impermeable to chlorine, and prolonged contact of the gas with chlorinated saw-dust has little effect in further removing lignin. The short method yields slightly more cellulose than does the long method, the difference between the two varying from 0·5 to 1·41 per cent. according to the kind of wood. In general, the pentosan content of the celluloses obtained by the two methods is about the same, and there is not much difference in the amounts of α-, β-, and γ-cellulose in the two products. Prolonged chlorination, however, breaks down the α-cellulose. W. P. S.

Estimation of Copper in Cellulose Substances by the Molybdomanganic Method of Fontès-Thivolle. H. Gault, B. C. Mukerji. (*Comptes rend.*, 1924, **178**, 711–13.)—The microchemical estimation of copper by the Fontès–Thivolle method, as applied to celluloses, gives very satisfactory results, and is carried out as follows:—Fifty c.c. of Fehling's solution in 100 c.c. of water are put into a pyrex glass flask provided with a mechanical agitator and a condenser ground into the neck. A known weight of cellulose (about 1 grm.) is introduced into the boiling solution, heated by a bath of calcium chloride at 120° C., and boiling continued for exactly 15 minutes. The cellulose, with the cuprous oxide, is separated by filtering and washing, and the cuprous oxide either dissolved directly in the phosphomolybdic reagent, or the filter paper and its contents are ignited in Pregl's micro-furnace, reduction to metallic copper effected by means of a current of pure hydrogen, and the copper dissolved in the reagent; the second method of procedure is twice as accurate as the former. The molybdenum is then estimated by titration with a 0·008 per cent. solution of permanganate. The whole estimation only takes 30 minutes. Tables of results for different purified celluloses and hydro-celluloses are given to three places of decimals, but it is pointed out that such precision is only of meaning when the different estimations are carried out under absolutely identical conditions. Corrections are necessary if other metals, particularly iron, are present. D. G. H.

Action of Trypsin on Various Leathers. A. W. Thomas and F. L. Seymour-Jones. (*J. Ind. Eng. Chem.*, 1924, **16**, 157–159.)—It is shown that trypsin is capable of hydrolysing collagen (*e.g.*, hide-powder) which has been treated with such substances as gallotannin, quinone, formaldehyde, and copper sulphate. Copper sulphate appears to combine with the carboxyl group, and in this case both tryptic and ordinary hydrolysis proceed to the same extent as with untanned hide-powder. With quinone and formaldehyde, combination probably takes place at the amino group, and the amount of hydrolysis depends on the quantity of tanning substance combined with the collagen. Some other kind of combination seems to occur in chrome tannage, since chromed hide-powder is not hydrolysed by trypsin.

W. P. S.

Inorganic Analysis.

Separation of Bismuth as Phosphate. G. Luff. (*Chem. Zeit.*, 1924, **48**, 61.)—The method previously described (ANALYST, 1923, **48**, 238) for the separation of bismuth from lead, copper, and cadmium has been found applicable to the separation from silver, manganese, zinc, nickel, cobalt, magnesium, aluminium, and ferric iron. Silver was recovered from the filtrate as chloride, and the five divalent metals as tertiary phosphates $M''NH_4PO_4$. Aluminium and iron were precipitated as phosphates by neutralisation of the filtrate with ammonia and digestion at boiling heat in a porcelain dish. The two precipitates, which are difficult to wash out, being contaminated with silica, must be evaporated with hydrofluoric and nitric acids until the weight after ignition is constant.

W. R. S.

Estimation of Alkali Metals in Aluminium and its Alloys. Schürmann and Schob. (*Chem. Zeit.*, 1924, **48**, 97–98.)—The method is based on the precipitation of aluminium chloride by saturation of the solution with hydrogen chloride. From 5 to 20 grms. of metal are dissolved in hydrochloric acid; the solution, diluted to 300 to 600 c.c., is cooled with a freezing mixture (ice-salt), and saturated with hydrogen chloride gas through a funnel dipping into the liquid. The precipitate is filtered off on a perforated porcelain disc covered with small porcelain fragments, supporting a 2 cm. layer of sand, which is again covered with porcelain fragments. The filtering material is previously extracted with hot strong hydrochloric acid until the extract leaves no fixed residue. The filter can be used again after being washed with dilute hydrochloric acid. The crystalline precipitate is washed with hydrochloric acid saturated with hydrogen chloride. The filtrate and washings are evaporated in a platinum dish, the residue is taken up with water and a few drops of hydrochloric acid, and the last traces of aluminium and iron are precipitated with ammonia. The filtrate is evaporated and treated with a few drops of ammonia and ammonium oxalate. The filtrate from the last operation is evaporated with sulphuric acid, and the residue ignited and weighed as sodium sulphate. This should be tested for purity by solution in water and addition of a few drops of ammonia and ammonium carbonate; if cloudy, the solution must be filtered, and the filtrate treated as before. Blank tests must be carried out, and the same glass vessels used in successive analyses. In the case of copper or zinc alloys, these metals are removed by the usual method before or after the precipitation of aluminium chloride. The process was found to be reliable for the separation of small quantities of sodium and lithium from large quantities of aluminium. (See also ANALYST, 1922, 47, 452.) W. R. S.

The Carrying down of Cobalt and Nickel by Tin Precipitated as Stannic Sulphide. V. Auger and L. Odinot. (*Comptes rend.*, 1924, 178, 710–711.)—Hydrogen sulphide, passed into solutions of stannic salts, generally results in the formation of mixtures of stannic sulphide and meta- and para-stannic acids, which are particularly liable to carry down cobalt and nickel. Solutions containing, respectively, about 24 mgrms. of cobalt chloride and 45 mgrms. of stannic chloride in 100 c.c. of water, acidified with hydrochloric acid, so that they contained 0·5 to 7 per cent. of hydrochloric acid (tin is not completely precipitated if more acid is present), were treated with hydrogen sulphide, and the precipitate washed with hydrogen sulphide water, and then dissolved in hydrochloric acid containing a few drops of nitric acid, evaporated to dryness, taken up with concentrated hydrochloric acid, and the cobalt estimated colorimetrically. The proportion of cobalt carried down was approximately in inverse ratio to the acidity of the solution, so that tin and cobalt cannot be separated by this method. If, however, the stannic solution is first reduced to the stannous condition, no cobalt is carried down. Nickel solutions behave in a similar way. If precipitation of the tin from the stannic solution is carried out by means of cupferron, no nickel or cobalt is found to be present.

Colorimetric Estimation of Cobalt.—Cobalt salts, dissolved in a large excess of concentrated hydrochloric acid, free from all traces of iron, give a blue coloration, due to formation of a cobaltohydrochloric acid. If increasing quantities of water are added to 40 per cent. hydrochloric acid, practically no lessening in intensity of colour occurs with 10 to 20 c.c. of water present in 100 c.c. of mixture, but, on adding more water, the colour diminishes rapidly, and when 7 c.c. of hydrochloric acid and 3 c.c. of water are present, is only 85 per cent. of its first intensity; 50 per cent. with 6 c.c. of acid and 4 c.c. of water, until finally, with 4·5 c.c. of acid and 5 c.c. of water, the colour turns pink, with re-formation of Co^{++} ions.

D. G. H.

Direct Estimation of Secondary Phosphate. I. N. Kugelmass and C. Rothwell. (*J. Biol. Chem.*, 1924, 58, 643–648.)—Saturated calcium sulphate is a precipitant of secondary phosphate, forming calcium orthophosphate according to the equilibrium equation: $4K_2HPO_4 + 3CaSO_4 \rightleftarrows Ca_3(PO_4)_2 + 2KH_2PO_4 + 3K_2SO_4$. This equation was confirmed by analysis of the precipitate, as well as by conductivity measurements. The following simple method is given for the direct estimation of secondary phosphate, containing at least 0·05 mgrm. of phosphorus as phosphoric acid in the presence of 20 times the amount of primary phosphate in solution, with an error of ± 5 per cent.:—To 1 c.c. of a solution containing at least 0·05 mgrm. of phosphorus as phosphoric acid in a centrifuge tube, 5 c.c. of saturated calcium sulphate are added, and the tube is put in a water bath at 60°

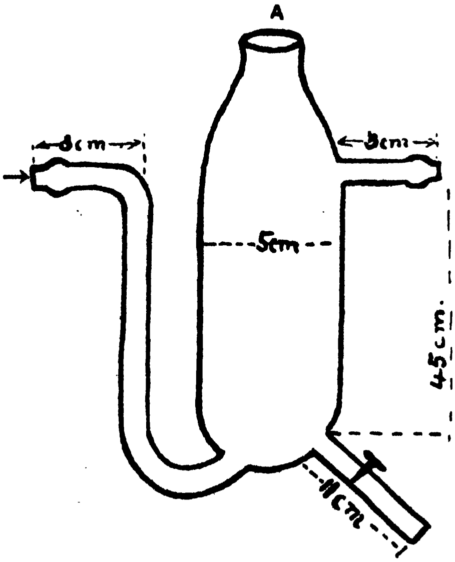
for a few minutes. The solution is centrifuged, the supernatant liquid is blown off, and the precipitate is twice washed with warm, half-saturated calcium sulphate solution. The final residue is dissolved in 10 per cent. nitric acid, the volume made up to 10 c.c., and the phosphorus is estimated in an aliquot portion by Briggs' modification of the Bell-Doisy method (*J. Biol. Chem.*, 1922, **53**, 13). The value of phosphorus obtained colorimetrically, multiplied by 2, equals the phosphorus of the secondary phosphate. Another method, very slightly different, is also given. The preparation of the materials is described. P. H. P.

Estimation of Sulphonitric and Sulphonitrous Acids. A. Glaire. (*Ann. Chim. anal.*, 1924, **6**, 40–41.)—The estimation of nitrogenous products in commercial and industrial acids cannot be accurately carried out by the usual methods, owing to reduction phenomena or formation of complex salts of nitrogen peroxide. Schloesing's ferrous chloride method for the estimation of acids and oxides of nitrogen gives, however, sufficiently accurate results for ordinary work. If an acid containing ferrous or cuprous impurities is introduced into the nitrometer a certain quantity of nitric oxide is formed, but a further proportion remains in combination with the metal. If the nitric oxide is gradually ejected, little by little, from the nitrometer by the side tube, till the acid reaches the level of the upper part of the apparatus, and the acid is run, drop by drop, with shaking, into a solution of permanganate, all the remaining oxides and acids of nitrogen go into solution. On treating this solution with ferrous chloride in the flask of Schloesing's apparatus, N_2O_3 is again reduced, and produces a further quantity of nitric oxide, which is measured. On examining an acid of known composition, it was found that these two volumes of gas together make up a total exactly corresponding to the nitrogen content of the acid. D. G. H.

Physical Methods, Apparatus, etc.

Fluorescent Powers of Cellulose and its Derivatives. S. Judd Lewis. (*J. Soc. Dyers and Colour.*, 1924, **40**, 29–41.)—An improvement in the author's method of spectro-fluorescometry is described, in which a rotating tungsten arc is used instead of a spark, as the source of light; this affords clearer and much more complete photographs. (The method has already been described, *cf. J. Chem. Soc.*, Abst., 1922, **122**, ii. 334.) When the fluorescent power is plotted against the wave length all the forms of pure cellulose exhibit the same type of curve, which is not materially affected by the physical condition of the fibres, whereas hydrocellulose, cellulose acetates, and oxycelluloses show definite and distinct forms, even in materials of quite different origin. It is suggested that the pronounced projections upwards and downwards, respectively, at wave-lengths about 2400 and 2900, and other similar features, indicate certain definite linkages which cellulose has in common with some of the sugars. The form of the curves, when compared with those of known cellulose derivatives, serves clearly to indicate the composition of a fabric or the progress of tendering operations. H. E. C.

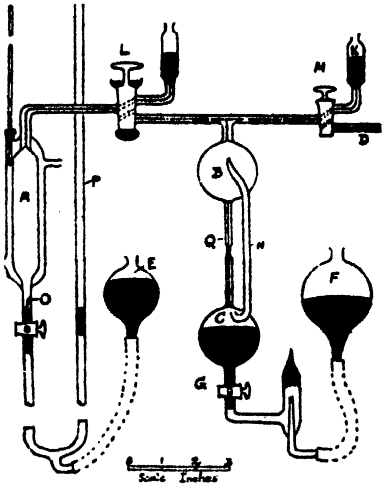
Improvements in Colorimetry. R. V. Stanford. (*Biochem. J.*, **1923, 17,** 839–843.)—It is not possible to compare the depths of colour of two solutions unless they are of the same shade of colour. Consequently the author (*Z. physiol. Chem.*, **1913, 87,** 159; Reports from the Chemical Laboratory, Cardiff City Mental Hospital, No. 3) described a dilution colorimeter, but it was inconvenient for actual use. In its original form the colorimeter has the usual pair of rhomboidal prisms, each of which illuminates one half of the field of view in the eye-piece. In front of the prisms there is a box containing two parallel-sided glass cells, one of which contains the standard solution, and the other the unknown solution. Over each cell is a burette, so that the solvent can be run into the stronger solution until equality of colour is reached. Improvements are now described which make this colorimeter as rapid and simple to manipulate as any other colorimeter. As an unvarying artificial source of light the Sheringham Daylight Lamp is recommended. Diagrams and a full description of the colorimeter, with an attachment to remove eye-strain and waste of time, are given. For a person of average perception in regard to colour about 3 minutes is the time required for a colorimetric comparison. This colorimeter has no errors due to principles. When equality is reached the liquids under comparison are of the same concentration, are being viewed through the same thicknesses of layer and, therefore, must be equal in all respects. The only trouble from the construction might be an unsymmetrical mounting, or a subsequent displacement, of the rhomboidal prisms. A mechanical stirrer is also described, and is an advantage, since a hand-stirrer is very fatiguing. P. H. P.



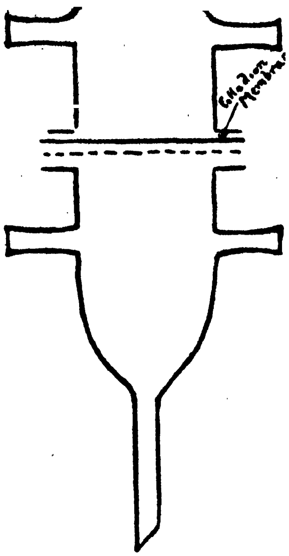
Apparatus for Drying Gases. V. T. Jackson. (*J. Ind. Eng. Chem.*, **1924, 16,** 163.)—A simple piece of apparatus for drying gases is shown in the illustration. A layer of glass-wool is placed at the bottom of the cylinder, and small glass beads are placed on the glass-wool to a depth of about 10 cm.; concentrated sulphuric acid is added until the cylinder is filled to a point about 1 cm. above the top of the beads, and the opening *A* is then closed with a rubber stopper. The gas is aspirated through the apparatus in the direction indicated by the arrow. If filled with potassium hydroxide solution, the apparatus may be used for removing carbon dioxide from air. The tapped tube at the bottom of the cylinder allows spent acid, etc., to be drawn off without disconnecting the drier from a train of apparatus.

W. P. S.

Absorption Pipette for Gas Analysis. S. W. Saunders. (*J. Chem. Soc.*, **1923, 124, 2826–2828.**)—The apparatus was designed to eliminate any joints between the measuring vessel and the absorption pipette. *A* is the water-jacketed measuring bulb forming one limb of a U-shaped manometer, the arrangement being for analysis at constant volume; the gas is confined between *L* and the pointer *O*, and its pressure measured. The bulbs *B* and *C* of the absorption pipette have about the same capacity as *A* (ca. 30 c.c. each); they are connected by a capillary constricted at *Q* and a tube *N* with a wide jet inside *B*. The apparatus is filled with mercury by raising *E* and *F*. After the gas has been introduced through *D*, the temperature and pressure are noted, and the reagent is sucked into *B* through glass tubing attached to *D*; no special connection is required. Any air introduced is expelled through *D*, mercury is allowed to run from *K* to *B*, and *M* is closed. The gas is transferred to *B*, and *L* is closed. If *F* is now raised, the reagent is sprayed through the gas *via N*. *F* is then lowered, after which the spraying is repeated. The gas is returned to *A*, followed through by mercury from *K*; the reagent is expelled through *D*, and the pipette washed with water. *B* is then filled with water and the gas washed and transferred to *A* for pressure measurement. The process is then repeated with other reagents. W. R. S.



Collodion Membranes of High Permeability. J. M. Nelson and D. P. Morgan, jun. (*J. Biol. Chem.*, 1923, **58**, 305–319.) The membranes here described are an improvement on those of Walpole (*Biochem. J.*, 1915, **9**, 287). The procedure for their preparation has been developed after many experiments. Four-inch circular glass plates were used. One, thoroughly cleaned to remove grease, rinsed with dust-free distilled water, dried in an oven at 50° C. and chilled if necessary, was weighed, carefully levelled on the desk and fogged, and 5 c.c. of a 2 per cent. solution of Du Pont's parlodion in 75:25 (by weight) alcohol-ether solvent mixture were run upon it and allowed slowly to evaporate. The plate was shielded from draught. As soon as a jelly formed the plate was removed to a balance and allowed to evaporate down to a weight found by trial to give, after water immersion, a grade (G_w), *i.e.* a "wetness" per grm. of dry collodion, somewhat higher than that desired. The water-wet membrane was gently removed from the plate under water and left to soak in water overnight. These membranes kept their grades for weeks. A few were dried in an oven to get the dry weight (D) which had to be known in order to calculate the (W_w), *i.e.* the weight of the membrane plus the water retained in the jelly, corresponding to the desired (G_w). $G_w = \frac{W_w - D}{D}$



The error in assuming that all membranes made from the same solution would have the same dry weight is, on the average, less than 2 per cent. The figure shows a filtration apparatus designed for the use of these membranes. Two pieces of heavy walled tubing, 2 inches in diameter, were flanged and ground to fit each other. By means of strong rubber bands over the arms the flanges were effectively held tightly together with the membrane between. Where necessary, the membranes were braced by the use of an ordinary nichrome gauze. Where they came in contact with the flanges the meshes of the gauze were filled with a gummy preparation consisting of rubber dispersed in paraffin to make the system air- and water-tight. This apparatus was set up in a suction flask connected with a manometer and a suction pump. These membranes were reasonably strong, and had a permeability almost three times as great as any previously reported. Their suitability for the study of invertase diffusibility has been established.

P. H. P.

Reviews.

The Electron in Chemistry. By Sir J. J. Thomson, O.M., F.R.S. Pp. 144. Philadelphia: The Franklin Institute. Price $1.75.

This volume contains the substance of five lectures given before the Franklin Institute in April, 1923, and which have since been published in the Journal of that Institute.

The object of these lectures was to show (1) that the electron is the dominating factor in chemical theory, and (2) the importance of interpreting chemical problems in the light of electrons and their orientation. How far this has been accomplished is doubtless a matter of opinion. Regarding the first point, the author has certainly succeeded, but in reference to the second the reviewer is at a loss to see what real advantage the chemist is to receive from the electron theory in its present stage of development.

The theory has had to suffer some extraordinary amendments in order to account for the existence of elements showing variable valencies, and for the trivalency of consecutive rare earths, although they have consecutive atomic numbers. It is true that some remarkable circumstantial evidence has been advanced in support of these amendments.

The character of the book may, perhaps, be obtained from an outline. Chapter I. is a discussion of the fundamentals of the electron theory, such as the configuration of the electrons and its relation to chemical properties. It is here that one has a good deal to accept. So far, it has not been possible to glean experimentally any information relating to the forces at play between the various components of the atom, and, in consequence, conjecture has had to be resorted to. In dealing with inter-electronic forces, the author writes, "I suppose that the repulsive force between two electrons is always inversely proportional to the square of the distance," but when the force between a positive nucleus and an electron is considered, an expression is assumed which would account for a force of attraction becoming one of repulsion when the distance between the charges is of the order of 10^{-8} cm. It is necessary to make this fundamental assumption, for we are told that "the mental picture conveyed by the multitude of orbits (of electrons, *i.e.* if the inverse square law held good) would be too blurred and complicated to be of much assistance in helping us to get readily a clear idea of what is going on in chemical processes." Later on, in referring to this vital assumption, he writes, "The size of atoms being what it is, is a proof that there is some law of physics not recognised in the older science which is all-important in connection with the theory of the atom, and must form the basis of that theory. If this law of force is that just given, then a number of electrons can be in stable equilibrium without necessarily describing orbits around it." From calculations of the size of atoms a curve has been constructed showing the relationship between the atomic diameters and atomic numbers. This curve is somewhat different from the well-known diagram of Lothar Meyer, inasmuch as the minimum diameters occur at the ends of the periods in the periodic classification and not in the middle.

Simple chemical combination is dealt with in Chapter II. Reasoning on the basis that eight is the maximum number of electrons which can exist in a layer of an atom in stable equilibrium not only leads to an explanation of the extended theory of valency of Abegg, but points to the existence of compounds not in accordance with these laws. "The electron theory states that any distribution of atoms and electrons in stable equilibrium is a possible compound, and will be saturated, provided that each electronegative atom is surrounded by a layer containing eight electrons." Then follows a discussion of the apparently important Polar and Non-Polar molecules, and also of the mathematical principles which have to be satisfied in any arrangement of electrons and atoms in a compound. Incidentally and in this regard, appears the trite statement that "chemistry is something more than freehand drawing." This chapter closes with a brief discussion of the disposition of electrons in typical compounds.

Combinations between molecules are studied in the third chapter. The treatment is based on Werner's Theory of Co-ordination—in spite of its serious defects—and the rôle played by polar molecules. The deduction is then made that electrolytic dissociation of simple and complex molecules in aqueous solutions is due to the influence of the polar molecules present. "Thus, in an aqueous solution of calcium chloride, the positively electrified part of the calcium atom would have next to it the negative ends of polar water molecules, and the attraction between it and the oppositely charged chlorine atom would be diminished."

This shows why calcium chloride will dissociate, and at the same time indicates why calcium ions must necessarily be hydrated.

The following chapter deals with the mechanism of chemical combination. Again, the part played by polar molecules is regarded as essential. The well-known experiments of H. B. Baker and H. B. Dixon, on the effect of moisture on chemical combination, are cited as being examples in which polar molecules in the form of water enable a reaction to take place. Had water molecules been present, they would have formed aggregates with the reactants, and union would have ensued, for the work, it is stated, required to separate the opposing aggregates is so much greater than that required to separate the elementary molecules. This is followed by a consideration of the influence of active surfaces in promoting combination, and of the formation of the colloidal "double layer," and once more, polar molecules play the important part. Among other topics, some attention is directed to Thiele's Theory of Partial Valencies, and to the production of light in the course of some reactions. Afterwards comes a comparative study of isotopes, the rare earths and transition elements, and elements showing variable valency. Those valencies which do not apparently conform with the theory are supposed to be caused by the wandering of one or more electrons into the inner layers, even though they already contain their maximum number. Accordingly, a certain dissymmetry is produced in the molecule, and this property seems to be confirmed by the fact that in such molecules the property of paramagnetism is pronounced.

The last chapter is devoted to an application of the electron theory to solids. Much support is forthcoming from calculations of such properties as "selective photoelectric effect" and compressibility of solids, and the surface tension when in the molten state.

There is a little mathematics scattered throughout the volume, but, as a rule, it is of an elementary nature. The book is well worth reading, and, coming from such an authority, should be read by all who are interested in the subject.

HUBERT T. S. BRITTON.

SYNTHETIC INORGANIC CHEMISTRY. ARTHUR A. BLANCHARD, Ph.D., and JOSEPH W. PHELAN, S.B. Pp. 321. New York: John Wiley & Sons; London: Chapman & Hall, Ltd. 3rd Edition. Price 15s. net.

The search for the best methods of teaching chemistry, or any other subject, will presumably continue till the end of time; each teacher has his own scheme, and fashions change in this as in other matters. Whereas, in organic chemistry, the preparation of various compounds constitutes the greater part of a student's practical work, in inorganic there is very little preparation work, and a considerable amount of time is usually devoted to analysis. The authors of this book are of opinion that greater stress should be laid on the preparation of inorganic substances, and they rightly call attention to the interest aroused by this type of experiment, as well as to the variety of manipulative processes involved. It cannot be denied that analysis by itself is too highly specialised to be adequate for the attainment of a general knowledge of chemistry and a course such as this book provides will doubtless prove of great value.

Of eleven chapters the first only is of a definitely quantitative character; the other ten are devoted to the preparation of typical compounds of the commoner elements in the various groups of the periodic classification, and experiments are included illustrating the properties of many of these compounds. A number of appendices at the end include solubility tables and other useful information.

This book reveals, once again, certain differences between American practice and our own. Many teachers will feel that there is in it a tendency to over-organisation and superabundant detail, which leave too little to the initiative of the student; on the other hand, these very features may be helpful to the teacher himself and his laboratory assistants, as a list of all the apparatus and materials required for each experiment will save time in preparing for the day's work. Three points may be selected as open to criticism: the frequent use of archaic names, such as blue and white vitriol; the introduction of new terms of doubtful utility, *e.g.* molal and formal solutions; the representation of the bleaching action of hypochlorous acid as $\text{color} + HOCl \longrightarrow \text{color oxide} + HCl$.

The printing and style of the book are satisfactory in every way.

A. F. KITCHING.

PLANT PHYSIOLOGY. By V. I. PALLADIN, edited by B. E. LIVINGSTONE. Second Edition. Pp. 360. Philadelphia: Blakiston, Sons & Co. Price $3·50.

This book is the second American edition of the authorised English edition of Palladin's "Plant Physiology." The first edition was based on the German translation of the sixth Russian edition, and on the seventh Russian edition of 1914.

The editor, Prof. Livingstone, of the Johns Hopkins University, U.S.A., deserves thanks for arranging this translation in 1917, for, as he says with justice in his preface to the first edition, "Its small size, together with its generally excellent arrangement and manner of presentation, render it very well suited to the use of beginning students, who really desire to obtain a general grasp of the subject in a comparatively short time."

The text of the 1922 edition is an improvement on that of the original translation, and the editor has added some new notes and a few fresh references. An important new feature, which will be of value to the student, is the introduction of a full summary of each chapter. For these the editor assumes entire responsibility.

To the chemist who is curious about the chemical happenings in a plant, this book may be recommended as a suitable introductory book to read, possibly in conjunction with *Practical Plant Biochemistry*, by Onslow, or *An Introduction to the Chemistry of Plant Products*, by Haas and Hill, or both. These books are cited in a long list of reference books classified by Dr. Livingstone, which list is a valuable adjunct of the book under review. Prof. Livingstone truly says, "Palladin approaches the subject from the point of view of a student of physiological chemistry, and it is the chemical aspects of plant physiology that here receive the greatest emphasis." A connected story is given of the synthesis of chemical materials in both green and non-green plants, and of the transformations and movements of these materials in plants. The energy requirements and transformations are discussed, and respiration in plants is analysed in terms of enzyme activity, in which analysis Palladin was one of the most noted pioneers.

The second part of the book, in which the vital manifestations of growth, reproduction, and plant movements are described, may be read with interest and profit, but it is not to be compared in worth with the first part of the book. There are few men who can at the same time satisfy the physicists, the chemists and the biologists of their time in text-books, either of animal or plant physiology. Sachs and Pfeffer in the past were able to do so, and their books on plant physiology are classics. With increasing knowledge the task becomes more difficult. The tendency to issue monographs written by specialists becomes more pronounced. This is to be desired, but a man who can put a connected story together will assist in realising a perspective otherwise difficult to obtain.

The specialist worker in plant physiology still feels this need, although this translation of Palladin's book is the latest important text-book in English; and, although the book has the value already indicated and has the additional value of containing a summary of his brilliant researches, it is somewhat behind the times. For example, none of the recent work on oxidations in the living cell is mentioned, and this work is all part of a chapter which Palladin helped to commence.

The writing of plant physiology monographs by experts has been, and will be, of great value to the advanced student, but there is still urgent need for a complete modern text-book of plant physiology.

M. THOMAS.

ORGANIC SYNTHESES. Vol. III. HANS THACHER CLARKE, Editor-in-chief. Pp. 104. New York: John Wiley & Sons; London: Chapman & Hall, Ltd. 1923. Price 7s. 6d. net.

The third volume of this series follows exactly the line of its predecessors, and, therefore, little need be added to what has already been said about Vol. I. (ANALYST, 1922, 47, 187) and Vol. II. (ANALYST, 1923, 48, 243); the general character is identical, and the same standard is well maintained.

It is satisfactory to notice that the number of contributors is steadily increasing, so that this book receives material from thirteen workers, in addition to the five members of the Editorial Board. There is growth, also, in the size of the volume, as the present number contains twenty-nine syntheses, extending over a hundred pages. The methods outlined are, of course, not new, but the endeavour all through has been to choose the best, and to work out in detail the most favourable conditions; special points are emphasised by notes at the end of each section. In six cases only are the yields claimed less than 60 per cent. of the theoretical.

The inclusion of a number of simple substances, such as acetamide and *p*-cresol is welcome, for it shows that improvement is possible even in long-established processes. It seems probable that careful revision of all the details of many elementary preparations would lead to results more satisfactory than is at present the case. The index covers all three volumes now published, and it is the intention of the editors to continue this collective index in subsequent volumes.

A. F. KITCHING.

COLLECTION OF LEGISLATIVE PRESCRIPTIONS CONCERNING CHEESE. By DR. A. J. SWAVING. Pp. 104. The Hague: Hugo de Grootstratt. 1923. Price 7s. 6d.

The present work, which is written in English, follows on the same lines as the author's *Recueil de Législations Beurrières et Margarinières concernant la Répression des Fraudes dans le Commerce du Beurre,* which was reviewed in the ANALYST some years ago (ANALYST, 1914, 39, 474). It will be invaluable to Public Analysts and others in this country who have to deal with the composition of cheese. The book contains the legislative enactments which are more or less in force in 33 different countries in all parts of the world, in many of which definite standards have been laid down for the composition, marking, and sale of various kinds of cheese. Apparently the compilation has been well and carefully done, and the book can be confidently recommended to those interested in this subject.

The position of the Public Analyst in this country, so far as the legal aspects of adulteration are concerned, is particularly unfortunate, and, in this connection, cheese is one of the most difficult subjects with which he is called upon to deal. A large quantity of skimmed, and partially skimmed, milk cheese is imported into this country from Holland, and is sold to the public as "cheese," although, in the country of origin, it cannot be so described. At the present time large quantities of such cheese, described by the makers as "half meat" and "three-quarter meat," are being imported into this country and are being sold to retailers as "Dutch Cheshire" and "Dutch Lancashire." The Ministry of Agriculture have been approached time and time again to make at least some efforts to prevent this gross misrepresentation, but, up to now, no action has been taken. There are rumours that the matter is again under consideration by the Ministry, and it may be that a perusal of that section of Dr. Swaving's book which deals with "The Dutch Cheese control under Government Supervision," will remove at least a portion of their apparent nervousness at issuing "cheese regulations."

G. D. ELSDON.

A SYSTEMATIC SURVEY OF RUBBER CHEMISTRY. (A bibliography, with copious abstracts, of the entire literature of rubber chemistry and closely allied subjects; thoroughly indexed by authors and subjects, with full cross-references, together with a patent index and introductory chapters summarising the present status of rubber chemistry.) By CLAYTON W. BEDFORD and HERBERT A. WINKELMANN. Pp. 385. New York: The Chemical Catalog Company. 1923. Price $7.00 net.

To the analyst who only occasionally comes in contact with the problems of, or related to, rubber chemistry, the work under review may be a little disappointing, for, notwithstanding the sub-title, a number of subjects, including Analysis of Rubber, Synthetic Rubber, Physical Properties of Rubber and Rubber Testing, have been designedly omitted, with the exception that articles dealing with these subjects have been included where they contain data relative to molecular structure, ageing, accelerators or similar subjects. The introductory chapters, both critically written, consist of articles on "Organic Accelerators of Vulcanisation," by Dr. L. B. Sebrell, and on "Theories of Vulcanisation," by Dr. W. J. Kelley. The summaries of our knowledge of these subjects, and the inferences drawn by the authors, should provide interesting and illuminating reading for a wide scientific public. The main object of the book is to present (with the exceptions already referred to), as accurately and as completely as possible, a systematic survey of the field of rubber chemistry up to January 1st, 1923. An excellent system of cross-indexing between authors, subject and patent indexes has been adopted, and the abstracts which accompany the references are designed to be indicative of the nature of the work rather than condensations of data.

A valuable feature of the references is that, in addition to one to the original, references are included showing the location of abstracts and reprints as well as of many discussions referring to the same in various books and reviews. For the benefit of future editions, the authors request the reader to make a note of all errors and omissions which he may observe, and it is to be hoped that Messrs. Bedford and Winkelmann's volume will receive in every direction the whole-hearted support which a work likely to prove of inestimable value to those interested in rubber science and technology undoubtedly deserves.

PHILIP SCHIDROWITZ.

CHEMICAL SYNONYMS AND TRADE NAMES. By W. GARDNER. Pp. 271. London: Crosby, Lockwood & Co. 1924. Price 25s. net.

One of the minor difficulties which consulting chemists have to surmount is the use of various trade names for one and the same product. This is particularly noticeable in the case of dyestuffs, for many of which, of the same composition, separate names have been devised by each firm of manufacturers or agents. There was, therefore, a distinct need for a concise dictionary of these trade synonyms, and the compiler may be congratulated upon the thoroughness with which he has done his work.

There are about 14,000 definitions of the chemical nature of trade materials, including, *inter alia*, dyes and pigments, drugs, explosives, minerals and alloys, and these are classified in alphabetical order, and comprise some 180,000 words. The system of cross references is very full; for instance, no fewer than 22 trade synonyms for adrenaline, and 16 for emerald green, are given, and each of these will be found in its proper place, with a reference to the key word.

It is a book which everyone who has to deal with trade chemicals in any way will find of the greatest value.

EDITOR.

MAY, 1924. Vol. XLIX., No. 578.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held on Wednesday, April 2, at the Chemical Society's Rooms, Burlington House. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. George William Clough, D.Sc. (Birm.), James Porter Shenton, F.I.C., and William Stanley Wood.

Certificates were read for the second time in favour of:—Messrs. John Joseph Bryant, Edgar Wilfred Deag, Harold Wilton Hewis, B.Sc. (Lond.), A.I.C., Ernest Wilfrid Jackson, F.I.C., Thomas McGrath, and Alfred Scholes, F.I.C.

The following were elected members of the Society:—Messrs. Philip Walter Alloway, Lorentz Oliver Brekke, B.Sc. (Leeds), A.I.C., Alex Munro Cameron, B.Sc., F.I.C., Thomas William Drinkwater, L.R.C.P., L.R.C.S. (Edin.), F.I.C., John Ralph Furlong, Ph.D. (Wurzburg), A.I.C., Ernest Griffiths-Jones, M.Sc. (Manc.), A.I.C., Basil Gordon McLellan, F.I.C., and William Thomas Rigby, F.I.C.

A Report on The World's Dairy Congress held at Washington, D.C., U.S.A., was made by John Golding, D.S.O., F.I.C., who represented this Society, and the following papers were read:—"The Routine Examination of Dairy Products with special reference to the Mojonnier Tester," by L. H. Lampitt, D.Sc., F.I.C., E. B. Hughes, B.Sc., F.I.C., and M. Bogod, B.Sc., A.I.C.; "Experiments on the Absorption of Copper following the Consumption of Vegetables containing Copper Sulphate," by J. C. Drummond, D.Sc., F.I.C., Miss M. G. Palmer, B.Sc., and Miss D. E. Wright, B.Sc.; and "Attempt to extend Mitchell's Colorimetric Method to the Catechol Tannins," by Miss P. H. Price, B.Sc. (work carried out under the Investigation Scheme).

The Composition of Beef and Malt Wine.

By G. D. ELSDON, B.Sc., F.I.C.

(Read at the Meeting, March 5, 1924.)

Some little time ago a sample of so-called beef and malt wine was submitted to the writer for analysis which was labelled in the following way:—"Liebig's Beef and Malt Wine. Made with Liebig's Extract of Beef and Malt Extract. Health, Strength, Vigour. A wineglassful may be taken two or three times a day. Non-exciseable." A small label on the neck bore the following words:—"This beverage is prepared in accordance with the requirements of the Food and Drugs Act and contains a small quantity of Salicylic Acid as a preservative."

As a result of the analysis of this sample, three formal samples were taken from the manufacturer in due course. The results of the analyses were so unsatisfactory that the samples were classified as adulterated and the one made the subject of proceedings was certified in the following way:

	Per Cent.
"Water	75·63
Total sugars	21·80
Alcohol	1·50
Other extractive matter	1·00
Salicylic acid	0·07
	100·00

This 'other extractive matter' contains 0·01 per cent. of nitrogen (calculated on the original liquid), which indicates the possible presence of not more than 0·2 per cent. of a mixture of equal parts of meat and malt extracts. This opinion is based on the fact that a mixture of equal parts of meat and malt extracts contains about 5 per cent. of nitrogen.

OBSERVATIONS.

No change had taken place in the article that would interfere with the analysis.

This is not a beef and malt wine. Its composition is similar to that of a flavoured cordial which usually contains up to 0·01 per cent. of nitrogen derived from sources other than beef or malt extracts.

The amount of salicylic acid present is contrary to the declaratory label on the bottle; it is six times that suggested as the maximum by the Departmental Committee on the preservation of food, 1901, in respect of beverages."

The vendors were prosecuted, and were fined the maximum fine in each of three cases, and 20 guineas costs were given against them. The analyses were accepted, the amounts of meat and malt extracts actually added being about half the maximum amount mentioned in the certificate. The contention of the defence was that it was impossible to market a non-alcoholic wine containing larger proportions of meat and malt extract than those present without the addition of a much higher percentage of preservative. Although the magistrate decided that this substance was not a beef and malt wine, the question arises as to what a beef and malt wine really is, and it may not be without interest to put before the Society the information which the writer collected while dealing with this matter.

Hutchinson, in his *Food and the Principles of Dietetics*, has the following passage:

> "*Medicated Wines* are concoctions, the basis of which is port or sherry, to which has been added extract of beef, extract of malt, peptone, pepsin, coca leaves, cocaine, cinchona, iron, or some other dietetic or medicinal substance. A 'beef and malt wine' may usually be regarded as containing about 1½ ounces of extract of meat and 2 ounces of malt extract in a pint of 'detannated' port or sherry. . . .
>
> The use of these wines can on no grounds be recommended. In the first place, they are not worth the price charged for them, for it is far cheaper and also better for an invalid to get beef or malt extract separately and take along with them, if need be, a definite quantity of sound wine of known antecedents. In the second place, it is open to grave question whether the ferment of malt (diastase) is not much impaired by the action of the alcohol to which it is exposed when dissolved in a fortified wine, such as port or sherry."

This seems a somewhat high standard, and is considerably higher than is given by any other authority that has been consulted. It is important, however, as a medical opinion, and is specially interesting when read alongside the analyses which follow, and the so-called professional opinion which often accompanies the wrappers on bottles of such wines.

Pharmaceutical Formulas, published at the office of the *Chemist and Druggist*, suggests that beef and malt wine should contain 4 ozs. of extract of beef and 8 ozs. of extract of malt per gallon of wine. This is equivalent to 2·5 per cent. and 5 per cent., respectively.

I have approached two manufacturers of beef and malt wine, and they state that they add 2½ per cent. of extract of beef and from 2½ to 5 per cent. of extract of malt; a standard which is practically the same as that given by *Pharmaceutical Formulas* above.

It would appear, therefore, that a beef and malt wine should be prepared by the addition of at least 2½ per cent. of beef extract and 2½ per cent. of malt extract, whilst the opinion of Hutchinson shows that these are most certainly minimum quantities. These quantities are, of course, the proportions which are added. A considerable proportion of these extracts is insoluble in water or is precipitated by the alcohol of the wine, so that the amount of nitrogen and phosphorus contained in the wine as it is finally delivered to the consumer will be substantially less than the amounts calculated from the proportions of material added. Experiments have been made to estimate the amount of nitrogenous matters which is so precipitated, and this matter is considered below when dealing with the question of standard.

For the purpose of estimating the proportion of beef and malt extract present the most valuable evidence is obtained, of course, from the estimation of the total nitrogen and of the phosphorus (P_2O_5) in the ash. These estimations are carried out in the usual way, viz. the total nitrogen by the Kjeldahl process, the phosphorus by precipitation as phospho-molybdate, solution of the precipitate in ammonia and reprecipitation with magnesia mixture. In order that there may be no loss of phosphorus during ignition it is better to add excess of sodium carbonate to the wine before evaporation to dryness, although in practice such loss probably very rarely occurs.

MALT EXTRACTS.—It is necessary to know the figures which are given by commercial meat and malt extracts; I have determined such figures on present-day samples and these are compared below with similar figures contained in the literature on the subject.

Published figures for the composition of malt extract are few. Penn ("Leach," 3rd Edition, p. 729), as a result of the analysis of three samples, gives the albuminoids as 3·1 to 4·9 per cent., the ash as 1·19 to 1·23 per cent., and the phosphoric acid as 0·43 to 0·57 per cent.

Rink (ANALYST, 1904, 29, 244) gives the ash of six samples as 1·34 to 1·64 per cent.

Harrison and Gair (*Year Book of Pharmacy*, 1906, p. 282), reporting on thirteen samples, give proteins varying from 3·6 to 7·0 per cent., but some of these samples were possibly adulterated.

The writer has examined several samples of commercial extract of malt purchased in the ordinary way by retail. These samples had the following composition, all figures being expressed on the original sample:

No. of Sample	Ash Per Cent.	P_2O_5 in Ash Per Cent.	Nitrogen Per Cent.
1.	1·4	0·57	0·99
2.	1·4	0·55	1·32
3.	0·88	0·36	0·73
4.	1·56	0·60	1·30
5.	1·60	0·70	1·46
6.	1·41	0·50	1·33
7.	1·31	0·34	1·20
8.	1·88	0·69	1·27

Sample No. 3 was a proprietary article specially prepared to obviate the awkward consistence of the ordinary extract; such an article would, of course, not be used in the preparation of a beef and malt wine. From these figures it would appear reasonable to take as averages for extract of malt, 1·3 per cent. of nitrogen and 0·6 per cent. of phosphoric anhydride.

MEAT EXTRACTS.—A number of different types of extract are on the market, but we need only consider the solid extract (originally prepared by Liebig, and of which "Lemco" is a typical example), as this is the material used by manufacturers of beef and malt wine—in fact, the wines are frequently termed "Liebig's

Extract of Meat and Malt Wines." The word "Liebig" in this sense usually bears no relationship to the Liebig's Extract of Meat Company (the original proprietors of "Lemco") and merely signifies an extract of meat made (or alleged to be made) by the process suggested by Liebig.

The following table gives the composition of various extracts according to different authorities.

Authority.	Ash Per Cent.	Phosphoric Acid Per Cent.	Total Nitrogen Per Cent.
Leach, 3rd Edn., p. 242	20·5—31·7	2·29—4·55	6·02—9·07
Hehner (Parry, "Food and Drugs," Vol. 1, 399)	18·8—29·4	5·16—6·95	8·21—9·80
American A.O.A.C.	Not more than 27		At least 8
König & Bomer	—	—	9·28
Elsdon	16·5—23·2	5·11—5·50	8·10—8·85
Villavecchia "Applied Analytical Chemistry," II. 16	17·0—25·0	—	8·5 —9·5

The last author states that not less than 56 per cent. of the extract should be soluble in 80 per cent. alcohol, and that the more usual value is 61 to 64 per cent.; this figure is used later in deciding what standard is reasonable for total nitrogen in a meat and malt wine.

WINES USED AS A BASE.—The wine which is generally used as a base in the preparation of meat and malt wine is a cheap Spanish or Portuguese wine of a "Port" character, but, of course, any cheap wine may be used. The following table gives results obtained for total nitrogen and phosphorus pentoxide by other workers on a variety of wines of different kinds:

Authority.	Ash Per Cent.	P_2O_5 Per Cent.	Total Nitrogen Per Cent.
König, Leach, 3rd Edit., p. 687	0·18—0·74	0·022—0·046	0·019—0·043
Bigelow, U.S. Dept. of Agri. Bur. of Chem. Bul. 59	0·05—0·45	—	0·014—0·147
Parry, "Food & Drugs," 1, 314	0·13—0·61	0·027* 0·014—0·068	—
Villavecchia, II. 220	0·15—0·45	0·02 —0·06	—

A number of wines of the type usually used by manufacturers—in fact, some of them have been wines actually taken from stocks kept for the purpose—have been examined by the author with the following results in grms. per 100 c.c.:

Sp. Gr.	Alcohol by vol. Per Cent.	Total Solids Per Cent.	Ash Per Cent.	P_2O_5 in Ash Per Cent. on original sample.	Total Nitrogen Per Cent.
—	15·9	—	0·23	0·027	0·021
1·0179	14·3	7·2	0·25	0·016	0·034
1·0172	16·2	8·3	0·32	0·044	0·083
1·0349	13·2	11·7	0·21	0·020	0·038
1·0372	13·5	12·6	0·26	0·013	0·025
1·0178	17·6	8·6	0·22	0·016	0·015
0·9954	9·2	1·6	0·29	0·048	0·024
0·9971	10·9	2·0	0·28	0·038	0·019

* Spanish red wine—average of many samples.

STANDARDS FOR BEEF (MEAT) AND MALT WINE.—From the various analyses given above it will be seen that the average figure for the total nitrogen in a meat (beef) extract is about 9·0 per cent., and that 8·7 per cent. is certainly not a high figure to take. Malt extract shows about 1·3 per cent. of total nitrogen, so that a mixture of equal parts of meat and malt extracts will contain about 5·0 per cent. of total nitrogen; this is the standard which has been adopted. In those cases where a meat extract containing a lower percentage of nitrogen than 8·7 has been used, the factor based on this percentage will not, of course, give the actual amount of extract added. This would not seem to be important, as it is immaterial to the consumer whether his wine is prepared from a small amount of a good extract or a larger amount of a poor one; it shows, however, the necessity of stating the percentage of nitrogen that has been assumed in order to calculate the meat-extract present.

Assuming, therefore, 5 per cent. of total nitrogen as an average figure for a mixture of equal parts of meat and malt extracts, and a minimum quantity of 5 per cent. of this mixture to be added to the wine, it follows that, assuming all the nitrogen to be retained by the wine, the prepared beef and malt wine will contain at least 0·25 per cent. of nitrogen, without taking into account the amount of this substance (some 0·03 per cent.) natural to the original wine. As mentioned above, however, a proportion of the meat and malt extract is not soluble in the wine, according to Villavecchia, about 40 per cent. being insoluble in 80 per cent. alcohol. Experiments by the writer have shown that about 70 per cent. of the nitrogen added as a mixture of beef and malt extracts remains in solution. It follows, therefore, that a wine sold as a beef and malt wine should contain at least 0·18 per cent. of total nitrogen. That this is not by any means a large amount is shown by the fact that some wines of a port character themselves contain up to 0·08 (or even more) per cent. of total nitrogen.

Treating the percentage of phosphorus pentoxide in the ash in a similar way, it will be seen that a mixture of equal parts of meat and malt extract will contain about 2·8 per cent. Using the same reasoning as for the percentage of total nitrogen, the finished beef and malt wine should contain at least 0·12 per cent. of phosphorus pentoxide, allowing 0·02 per cent. for the amount naturally present in the wine, and assuming (as has been shown by experiment) that about 70 per cent. of the total phosphorus pentoxide of the meat and malt extracts goes into solution.

COMMERCIAL SAMPLES OF MEAT (BEEF) AND MALT WINE.—In order to discover how far various commercial samples could be truthfully described as "Meat and Malt Wine," a number of these have been examined, and the results are given in the following table. Figures obtained from an examination of various proprietary wines which are supposed to be of a beef and malt wine character are also included. Comment is hardly necessary, but it is interesting to observe that sample No. 8 had a label attached which bore the following words:—"The presence of a LARGE PROPORTION OF PHOSPHATES is distinct evidence of the presence of both malt and beef, while the IMPORTANT QUANTITY of NITROGENOUS EXTRACTIVES affords further confirmation."

No.	Sp. Gr.	Alcohol by volume Per Cent.	Total Solids Per Cent.	Ash Per Cent	P_2O_5 in Ash Per Cent.	Total Nitrogen Per Cent.	Description.
1.	1·0505	18·0	16·7	0·32	0·060	0·082	Liebig's Beef & Malt Wine
2.	1·0257	19·2	10·7	0·53	0·140	0·115	„
3.	1·0367	16·1	7·4	0·54	0·090	0·110	„
4.	1·0490	16·3	15·4	0·38	0·094	0·098	„
5.	1·0494	20·4	16·6	0·32	0·048	0·052	„
6.	1·0280	19·2	9·6	0·54	0·100	0·040	„
7.	1·0234	18·0	9·7	0·27	0·018	0·120	Tonic Wine
8.	1·0286	17·6	10·2	0·32	0·047	0·052	——'s "——"
9.	1·0326	17·1	10·5	0·26	0·038	0·040	——'s Wine
10.	1·0341	17·4	11·3	0·33	0·062	0·058	"——"
11.	1·0262	16·0	8·7	0·36	0·060	0·049	"——"
12.	1·0530	18·6	16·2	0·44	0·080	0·080	——'s "——"

The Volumetric Estimation of Columbium.

BY W. R. SCHOELLER, Ph.D., AND E. F. WATERHOUSE.

THE volumetric estimation of columbium is based on the reducibility of columbic acid in solution and the re-oxidation of the lower oxide by permanganate, tantalic acid being unaffected under the same conditions. Assuming quantitative reduction to the sesquioxide, then, according to the equation $Cb_2O_3 + 2O = Cb_2O_5$, each c.c. of 0·1 *N* permanganate indicates 0·006655 grm. Cb_2O_5 (Cb = 93·1).

Two methods for the volumetric estimation of columbium have recently been published. The first is that of Metzger and Taylor (*J. Soc. Chem. Ind.*, 1909, **28**, 818), in which the mixed oxides of tantalum and columbium are fused with bisulphate; the melt is dissolved in sulphuric acid with addition of succinic acid to prevent hydrolysis, and the hot solution passed through a Jones reductor containing amalgamated zinc. The reduced solution is received in a flask filled with carbon dioxide and titrated with 0·1 *N* permanganate, one c.c. of which equals 0·007052 grm. Cb_2O_5.

The other method, due to Levy (ANALYST, 1915, **40**, 204), is a modification of that of Osborne (*Amer. J. Sci.*, 1885, **30**, 328). The mixed oxides are dissolved in hydrofluoric acid; the bulk of the acid is evaporated, hydrochloric acid is added to the remainder, and the solution reduced with granulated zinc in a flask filled with hydrogen. When all the zinc is dissolved, the liquid is diluted with sodium phosphate solution and sulphuric acid and titrated with 0·1 *N* permanganate. One c.c. = 0·00833 grm. Cb_2O_5 (Osborne's factor is 0·00763).

The main object of the present paper is to call attention to the unreliability of the volumetric estimation of columbium by the methods outlined, the authors' conclusions being drawn partly from their own work, partly from that of Treadwell (*Helv. Chim. Acta*, 1922, 5, 806; ANALYST, 1922, 47, 533).

The above factors show that neither process achieves a stoichiometric reduction, and that the sesquioxide stage is much more nearly reached by the powerful action of a zinc reductor than by simple solution of granulated zinc in the acid liquid. The supposition that definite intermediate oxides, such as Cb_3O_5, Cb_8O_{13}, $Cb_{10}O_{17}$, etc., are formed in the reduction may be dismissed as unlikely until their existence can be proved; no doubt the correct view will prove to be that only part of the pentoxide undergoes reduction, the balance remaining unaffected. Now, the extent of such incomplete reaction is not likely to be exactly the same every time, being easily affected by slight changes in the conditions under which it is carried out. Hence it is not surprising that the degree of accuracy attained in either process is admittedly not very great.

A. METZGER AND TAYLOR'S PROCESS.—A satisfactory explanation of the incomplete reduction of columbic acid in this process was given by Treadwell (*loc. cit.*), who found that columbium solutions prepared with addition of succinic acid are unstable, as they deposit a white precipitate on standing, and that their reducibility decreases with increasing age or dilution. On titrating the reduced solutions he obtained irregular results, and arrived at the conclusion that columbic acid is present partly in the colloidal state, only the dissolved portion being acted upon by the zinc. To verify this inference, he titrated columbium solutions in presence of titanic sulphate, ammonium molybdate, or ammonium vanadate in known considerable excess, and obtained at once a more nearly complete and constant reduction. The addition of these salts produced soluble complexes in which the columbic acid was reduced completely; but Treadwell could not calculate the extent of the reduction, as he had no pure columbium pentoxide at his disposal.

A short time prior to the appearance of Treadwell's paper the authors had concluded an investigation, begun in 1920, into the volumetric estimation of columbium. This comprised a repetition of the work of the earlier investigators and the full elaboration of a process suggested in the last paper published by the late W. B. Giles (*Chem. News*, 1909, 99, 1). In the test analyses, by Metzger and Taylor's method, the fusion of columbium pentoxide with bisulphate was carried out in glass flasks, after addition of 2 to 3 c.c. of sulphuric acid, which obviated the need of using platinum vessels; otherwise the original directions were carefully followed, particularly as regards amalgamation of the zinc. Four titrations carried out with +30-mesh amalgamated zinc (ordinary pure) gave a mean factor of 0·007424 grm. Cb_2O_5 per c.c. of 0·1 *N* permanganate (maximum deviations, +0·77 and −0·89 per cent.), while two tests, in which chemically pure zinc granules (similarly amalgamated) were used, gave 0·008139; we were thus unable to get the same factor as Metzger and Taylor's (0·007052). Our results exemplify the magnitude of the error likely to arise, not only from the indiscriminate use of the same factor by independent workers, but even from a change in the supply of zinc; they satisfied us as to the remarkable indefiniteness of the reaction of metallic zinc upon columbic acid, to which the disparity between Levy's and Metzger and Taylor's factors had already pointed. Metzger and Taylor found the degree of reduction to vary even according to the extent to which the zinc had been amalgamated, and found it necessary to prescribe definite proportions of zinc and mercury for the amalgamation. Treadwell's comment is that the cause of the influence of the amalgamation on the degree of reduction of the columbic acid is not elucidated by the experimental evidence.

B. Osborne and Levy's Method.—Treadwell investigated the reduction of columbium fluoride dissolved in strong hydrochloric acid, but not by addition of granulated zinc as directed by Levy; he passed the solution through a cadmium reductor and titrated it with permanganate in presence of manganous sulphate. Proceeding in this manner he obtained concordant results, but could not arrive at a factor as his columbium preparation was contaminated with tantalum. Treadwell observes that if the hydrofluoric acid solution is evaporated to dryness and the residue taken up in strong hydrochloric acid the resulting solution is not always clear, while any excess of hydrofluoric acid not expelled by evaporation prevents complete reduction. We desire to emphasise this point, as it forms one of our arguments against Levy's method.

In our test analyses, ten estimations were made in strict accordance with Levy's directions. Nine titrations gave an average factor of 0·01006 (Levy's factor: 0·00833), with maximum deviations of +3·75 and −5·30 per cent. We ascribe these errors chiefly to the interference of hydrofluoric acid, the removal of which "*nearly* to dryness" (Levy's italics: original, p. 208) is, in our opinion, a great weakness of the method; for "enough hydrofluoric acid must be present to give a perfectly clear solution in hydrochloric acid," but "the quantity must be so small as not to interfere with the reduction" (p. 209). We argue with Treadwell that any quantity of free hydrofluoric acid, however small, prevents the reduction of a corresponding quantity of columbium. We must assume our manipulations to have been made correctly, for in all cases we obtained clear solutions in hydrochloric acid and observed the formation of a bulky white precipitate within two minutes after the end of the titration. Levy says: "If this precipitation does not take place the result obtained should be treated with suspicion, probably being too low" (p. 210).

A source of error not investigated by us was pointed out by Levy himself in the variation of the tantalum present, low results being obtained with low ratios of columbium to tantalum. This can be explained by the formation of fluoride complexes, tantalum fluoride acting like an excess of hydrofluoric acid in preventing the reduction of part of the columbium.

A most serious objection against the three methods investigated is the influence of the physical and chemical state as well as the quantity of the reducing agent. We used the same brand of chemically pure, granulated zinc in the work on Levy's method; his original directions prescribe the use of shot "23 of which usually weighed 10 grms." We conformed as closely as possible to this specification except in the last test, in which 10 grms. of coarser shot was used, with the result that reduction was now less complete, as indicated by the factor 0·01124. Levy, on the other hand, found less complete reduction to attend the use of very small pieces, which dissolved too quickly; we conclude that the absence of a zinc reductor is an additional point against the method, being the cause of less complete and uniform reduction.

C. NEED OF STOICHIOMETRIC REDUCTION.—The evidence given in the preceding paragraphs shows that the volumetric estimation of columbium in its present form lacks the features of a reliable oxidation process. In the volumetric estimation of iron the reduction may be carried out with hydrogen sulphide, sulphur dioxide, stanuous chloride, or zinc (either in Jones' reductor or added to the solution); the iron and acid concentration may vary within wide limits, and the presence of a large number of other elements is without influence: the same factor is always valid, and it is obtained by stoichiometric calculation. In the case of columbium the factors are quite empirical; not only were we unable to get those obtained by Metzger and Taylor and by Levy, in spite of close adherence to their directions, but every time a slight variation in the working conditions occurred, a serious deviation in the factor was the result. Observations to the same effect were made by Treadwell as well as by the authors of the methods under consideration. We are forced to the conclusion that the two processes must be regarded as unreliable.

Speaking of his method, Levy says: "Reduction does not go as far as in any of the others, which may be an advantage, in that the less one aims at, the more likely one is to obtain that little" (p. 209). With the practical experience gained, we totally disagree with this view, and consider that, in an accurate oxidation method, reduction should always proceed to a definite oxide regardless of slight variations in the working conditions and the amount of tantalum present. In the volumetric estimation of other elements, we know of no reliable method, in use at the present time, that is not based on stoichiometry.

Treadwell points out that the immediate difficulty in the case of columbium consists in obtaining the whole of the element in a state of true molecular solution, and as he has intimated his intention to continue his experiments in that direction, the authors have suspended further work for the present.

A brief consideration of the reduction of the oxides of two allied metals will support the authors' views on the necessity of stoichiometric reduction: tungstic, like columbic acid, does not give true solutions with strong acids. When the solution of tungstic acid in hydrochloric acid is reduced with zinc a colloidal blue precipitate is formed. The re-oxidation of the lower oxide gives erratic results, and there is no satisfactory oxidation method for tungsten. Molybdic acid is readily soluble in strong acids, and after passage through Jones' reductor the molybdenum in this brown solution can be estimated accurately with permanganate, reduction proceeding quantitatively to the sesquioxide. In feebly acid solutions, however, molybdic acid exists in a form more akin to that of tungstic and columbic acids, and such solutions upon reduction furnish colloidal molybdenum blue. This is of indefinite composition, intermediate between the tri- and the sesquioxide.

D. GILES' PROPOSED METHOD.—Giles (*loc. cit.*) suggests the use of zinc dust as a reducing agent, and effects solution of columbium pentoxide by fusion with potassium carbonate, solution in water, and boiling with excess of phosphoric acid until the solution is quite clear. The last paragraph of his paper gives a short outline of the process he intended to work out; but no further paper was published by him. We desired to ascertain whether the intensely-coloured solution produced by the energetic action of zinc dust upon the phosphoric acid solution of columbium might not contain the whole of that element as sesquioxide. Our results prove that such is not the case; no useful purpose would therefore be served by a detailed account of our method, the numerous tests made, and the elaborate apparatus devised, in which addition of zinc dust, reduction and heating, filtration through a quartz-sand column, washing, and collection in a solution of ferric alum were carried out under complete exclusion of air. The following is a *résumé* of our observations, which all confirm what has already been said concerning the erratic behaviour of columbic acid in contact with zinc:

(1) Eleven consecutive titrations gave a mean factor of 0·006990, with maximum deviations of +1·00 and −1·91 per cent., hence reduction stopped short of the sesquioxide. In these tests 5 grms. of zinc dust were used for 0·25 grm. of columbium pentoxide; the solution was obtained according to Giles' suggestion, no bisulphate being employed.

(2) When the quantity of zinc dust was doubled, reduction was less complete, the increase in zinc being equivalent to a decrease in the acid concentration: the mean factor from six titrations was 0·007261 (maximum deviations, +2·25 and −2·22 per cent.).

(3) If the zinc dust in the above method was replaced by pure zinc powder, filings, or granules, the results were altogether hopeless; the burette readings registered between 10 and 80 per cent. of the reduction attained with zinc dust. Magnesium was also tried: the reduction was almost nil.

(4) In the final series of tests, the columbium pentoxide was fused with bisulphate and sulphuric acid, the cold liquid heated with phosphoric acid, and the solution reduced with zinc dust, etc., as before. Entirely different values were now obtained; for tests in which 5 grms. of zinc dust were used, gave an average factor of 0·008365 (maximum errors, +2·19 and −2·48 per cent.), while two tests with 10 grms. of zinc dust gave 0·009897.

In conclusion, the authors' modification of Giles' method must also be regarded as unreliable; under strictly uniform conditions fairly concordant results may ensue, but discrepancies occur as in the other methods, and a slight change in the working conditions causes serious divergences in the degree of reduction. The factors obtained are non-stoichiometric. The presence of a colloidal phase is, no doubt, the cause of the irregularities.

SUMMARY.—(1) Theoretical considerations and the results of original tests are submitted to prove that the existing methods for the volumetric estimation of columbium are unreliable. These methods furnish empirical factors.

(2) The view is advanced that the criterion of an accurate volumetric method should be a stoichiometric factor, indicating reduction to a definite oxide.

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Apparatus for Extraction and Solvent Recovery.

BY S. A. DE LACY, A.I.C.

(*Demonstrated at the Meeting, February* 6, 1924).

IN systems of extraction by volatile solvents the vessel containing the solution of the extracted material is removed and connected with a condenser, so that the solvent may be recovered by distillation.

This apparatus has been devised to obviate the inconvenience of disconnection with its attendant losses of time, solvent, and heat. In it, the container of the substance to be extracted is suspended in the extraction vessel so that the condensed solvent from a reflux condenser drops immediately into the upper end of the container, percolates through the substance and, charged with extracted material, eventually falls into the boiling liquid.

The essential feature of the apparatus consists of a glass frame operated by a key external to the extraction vessel, by which means the reflux tube or distillation tube may be closed. The frame consists of a thick circular rod S, having near its upper end two parallel surfaces cut; to its lower end an oval platform P is attached with a drip point D beneath it. The support for the container of the substance to be extracted is attached to the lower surface of the platform by the upright U so that the apex of the drip point lies over the centre of the container, the junction of the upright with the platform being separate from the drip point, since otherwise solvent tends to run down the upright instead of falling from the drip point.

The support for a folded paper, made by shaping ashless filter paper on a thick piece of plate glass and securing the turned up end, is shown in Fig. 1. This form has two arms, A_1, A_2, attached to U. In the oval arm A_1 there is a gap through which the upper end of the paper is inserted until it fills the arm, when in position; the lower end of the paper drops within the closed rectangular arm A_2 on to the cross bar J.

In Figs. 2 and 3 the distinct forms of support hold the container by a strong glass pin passing through holes placed in its sides so that it is vertical when its weight rests on the pin; they are specially suitable for alundum extraction thimbles. The substance may be covered by a perforated disc of filter paper or some cotton wool, the pin being above this.

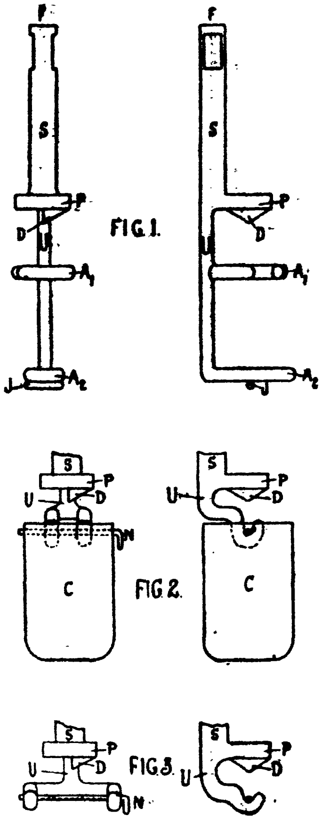

FIG. 1.

FIG. 2.

FIG. 3.

ELEVATION, FRONT AND SIDE.

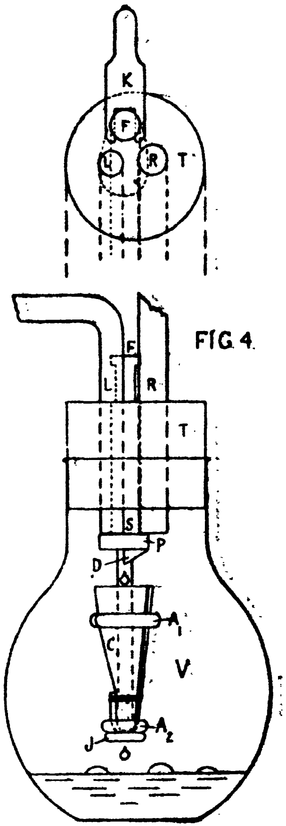

FIG. 4.

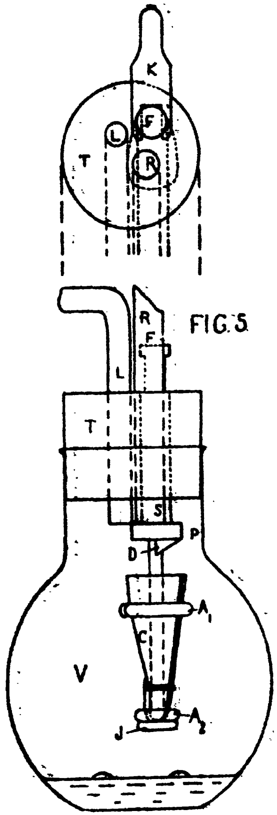

FIG. 5.

DIAGRAM OF FRAME AND ITS USE.
(Similar letters refer to similar parts throughout).

ELEVATION AND PLAN.

ELEVATION AND PLAN.

Fig. 2 shows a support having the extremities of its arms projecting inside the container and bent to form vertical slots. In use, the upper end of the container is placed over the extremities, then a pin N is pushed through the holes in the container sides to rest easily in the slots.

Fig. 3 illustrates a support composed of a horizontal semi-circular arm, the ends of which are bent to form vertical slots. In use, the holes in the container sides have a pin pushed through them and its projecting ends are dropped into the slots as the container is placed within the arm. All supports are made of glass rod, making the frame capable of hard wear.

The stopper of the extraction vessel (in this instance of cork) has three holes in it, and the frame end F is pushed through the suitable orifice in the base of the stopper until the flat surfaces are just clear of the upper surface of the stopper when a rectangular key is fitted on them with its lower surface resting on the stopper and its upper surface against the uncut portion of the rod S. A reflux condenser tube and a tube L, to conduct away the vapour of the distilled solvent, are pushed down through the remaining holes until the flat ends just clear the upper surface of the platform. The container is placed in the support and the platform moved into the position for extraction, that is, with the portion above the drip point partially closing the orifice of the reflux condenser tube, while the tube L for distillation is shut off (Fig. 4). The vessel containing the solvent is connected and the solvent boiled, when the condensed vapour runs down the reflux tube on to the platform, over its edge, and falls from the drip point D. After extraction is considered complete the platform is moved to close the tube R while the tube L is left entirely free; thus the solvent is recovered (Fig. 5). No solvent falls from the drip point in this position, so that, on disconnection, the extracted material is in the vessel, and the dry container, with its unextracted contents, can be easily removed. In both the above positions of the frame the condensed solvent on the upper surface of P forms a liquid seal for a completely shut-off tube, and in no intermediate position does P close both tubes; this ensures safety. An indifferent gas may be passed through the apparatus during the operations.

When difficulty in extraction is anticipated the excellent method of Stokes (ANALYST, 1914, 39, 295) may be employed. The platform being as in Fig. 5, sufficient solvent is placed in the extraction vessel to cover the substance; thus the warm liquid asserts its soaking action while the solvent slowly distils. The frame is then turned so that extraction may be finished as in Fig 4, after which the remainder of the solvent is recovered by the return of the key to its original position.

Where no container is held in supports, as in Figs. 2 and 3, it is obvious that the system may be used with advantage for reactions which require boiling under a reflux condenser, followed by the subsequent distillation of unaltered solvent or a resultant product.

The bench and permanent wall-type forms of the apparatus demonstrated were devised and constructed by the author. For modifications and further details see Eng. Pat., 206, 711, Nov. 22, 1922, and addition 28,018, Nov. 7, 1923.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

NOTES ON THE LIQUID FROM RIPE COCONUTS.

THE liquid from ripe coconuts varies from nearly clear to distinctly opalescent; in the latter condition, when examined under the microscope, it shows fatty globules, agglomerations of fat and amorphous matter. No starch granules or anything staining blue with iodine are seen. One or two rod bacteria are present.

The liquid from one nut (No. 4) was measured and found to be 122 c.c. It, like the other three examined, had the usual sweet sappy taste, with the characteristic nutty flavour.

Specific Gravity.	Water = 1000.	Solids in grms. per 100 c.c. (Sp. gr. - 1000). ÷4·25.*
Mixed liquid from 2 nuts	1042·1	9·92
Nut 3	1055·5	13·06
Nut 4	1045·9	10·80

Average sp. gr. =1047·85. Average solids =11·26 g. per 100 c.c.

Optical rotation of the 3 samples of liquid reading 100 mm. tube Soleil-Ventzke-Scheibler instrument:

	AD 4·25. div.
Nos. 1 & 2	$a=10{\cdot}6=37{\cdot}0$
3	$a=16{\cdot}1=42{\cdot}7$
4	$a=13{\cdot}2=42{\cdot}3$

Average specific rotatory power =40·7

Further experiments were made with the liquid from Nut 4. The amount of ash obtained was 1·25 grms. per 100 c.c.

It had some reducing effect with Fehling solution, 1 c.c. giving 0·011 CuO =1·1 grm. per 100 c.c., which brought to invert sugar or dextrose by divisor 2·31 =0·47 per cent. of reducing sugar.

A dilution of the liquid, 10 c.c. to 100 c.c. with distilled water+0·5 grm. of citric acid, when digested at boiling temperature for one hour gave a greatly increased reduction with Fehling solution. Thus 2 c.c. gave 0·071 CuO =17·75 grm. of CuO per 100 c.c. of original liquid, which, corrected for the original reducing power and brought to reducing sugar (invert) by use of the divisor 2·31, gave 7·21 grms. per 100 c.c. of invert sugar =6·87 original sucrose from which it proceeded.

The change in optical rotation caused in the above experiment was calculated into sucrose and gave 7·3 grm. per 100 c.c.

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[*] *Dry Solids*:—10 c.c. of (Nut 4) dried in a platinum dish in water oven and weighed at intervals till a point of fair constancy was reached before definite browning set in gave 1·08 grms. = 10·8 grms. per 100 c.c. (Sp. gr. 1045·9 - 1000÷10·8=4·25 divisor for solids).

The coconut liquid (No. 4) proved to be directly fermentable by Burton yeast;[*] at the end of an active fermentation the difference between the original gravity and the extract gravity was 28 degrees; this divided by 3·86 (the solution divisor for sucrose) and corrected for the 0·47 grm. per 100 c.c. of reducing sugar originally present, gave 7·1 grms. per 100 c.c. fermented, which, in face of the evidence afforded by the citric acid inversion, may be confidently regarded as sucrose. We thus get:

	Grms. per 100 c.c.
Sucrose by inversion and CuO	6·87
,, ,, optical rotation	7·30
,, ,, fermentation	7·10
Average	7·09

When the liquid is warmed coagulation sets in at about 43° C. and becomes more and more pronounced as the temperature rises towards boiling point. The white coagulum was separated by filtration and examined. It gave evidence of containing cellulose in some form, a portion being soluble in ammoniacal cupric oxide, reprecipitable on rendering the solution slightly acid, and it showed nitrogen by Armstrong's test (heating with sodium, etc).

The original liquid tested with paste made from potato starch showed a feeble diastatic power only, the paste becoming somewhat thinner. The approximate P_H value of the original liquid was 6·0

There can be little doubt that the coconut liquid (or milk, as commonly so-called) is a very complex and interesting liquid, containing, as it must do, the substances necessary to the deposition or elaboration of the flesh of the nut. Cellulose matter and fat evidently come from it; and it is a fair deduction that sucrose plays a very important part in the cell metabolism.

C. G. MATTHEWS.

THE LABORATORY, BRIDGE CHAMBERS,
BURTON-ON-TRENT.

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[*] The fermented liquid had a poor, vapid taste.

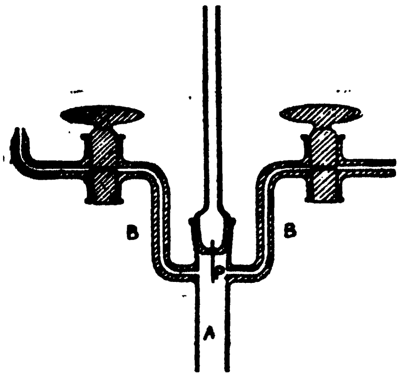
HYDROGEN ELECTRODE FOR USE WITH MEAT PASTES, ETC.

A KNOWLEDGE of the P_H value of meat pastes, potted meats and fish, etc., is sometimes useful as indicating the liability of these products to become infected with food poisoning organisms, more especially *Bacillus botulinus*. This organism is unable to develop in media the acidity of which exceeds a certain point.

With the form of electrode here figured, these determinations may readily be carried out on the meat paste itself, without previous extraction with water.

The central tube A is tightly packed with the meat paste, and, with the aid of a small glass plunger, the upper surface of the paste is levelled off, so as to be just in contact with the tip of the platinum wire P. The lower end of the column of meat paste is flush with the bottom of the tube A. The electrode is then adjusted so that the lower end dips into a beaker containing saturated potassium chloride solution, and the electrical circuit joined up in the usual manner (ANALYST, 1921, 46, 318). A slow stream of pure hydrogen (preferably electrolytic) is passed continuously through the tubes B.B while the readings are being taken. If the meat paste is tightly packed, its conductivity is quite high enough for sharp readings to be obtained on the bridge.

Several samples of potted meat and fish examined in this way gave the following P_H values:



			P_H
Chicken, ham and tongue	Sample	1	5·33
		2	5·45
Chicken and ham	,,	1	5·58
	,,	2	5·60
Ham and tongue	,,	1	5·60
	,,	2	5·65
Salmon and shrimp	,,	1	5·82
	,,	2	5·82
Salmon and anchovy	,,	1	5·55
	,,	2	5·63
Shrimp	,,	1	6·28
	,,	2	6·43

G. W. MONIER-WILLIAMS.

MINISTRY OF HEALTH,
WHITEHALL, S.W.

TESTS FOR THE PURITY OF CARBON TETRACHLORIDE.

IN view of the recent extensive use of carbon tetrachloride as a remedy for hook-worm, and the occasional reports of serious symptoms arising after its administration, some investigations into the tests for the purity of this drug were undertaken. Most of the tests which could be found in the books available were tried, and those which were found most useful are detailed in this paper, which is offered for publication in the hope that other analysts may be induced to give their experience of such tests, and also their opinion as to the standard of purity to be demanded before any sample of this drug is passed for use as medicine.

The tests proposed are:

I. FOR ADULTERATION: THE DENSITY.—This was determined on three specimens, which all appeared to be pure by other tests, and was found to be, at 30° C.

$$1{\cdot}5744 \pm 0{\cdot}0001$$
$$1{\cdot}5745 \pm 0{\cdot}0005$$
$$1{\cdot}5742 \pm 0{\cdot}0003$$

Between 15° and 30° C. the rate of change of density with temperature was found to be $0{\cdot}00200 \pm 0{\cdot}00005$ per degree, and linear within this error. The density at 15° C. is, therefore, $1{\cdot}6044 \pm 0{\cdot}0007$. These figures are true densities, *i.e.* the mass in grms. of 1 c.c. of the liquid.

II. FOR PHOSGENE, ETC.—Some of the carbon tetrachloride is shaken with water in a separating funnel and the water phase tested for

(1) Neutrality; (2) absence of chlorides; (3) absence of free chlorine.

The benzidine test was also used as for chloroform, but, as benzidine is insoluble in carbon tetrachloride, about 10 per cent. of chloroform was added to bring the benzidine into solution. Obviously a blank test with the chloroform used must be made simultaneously.

III. For Carbon Bisulphide.—One c.c. of the carbon tetrachloride is mixed with 2 c.c. of aniline oil and 2 drops of ammonia solution (0·880). After standing five minutes 1 or 2 c.c. of a freshly made, dilute solution of sodium nitroprusside (about 1 per cent.) are added, and the mixture shaken. A purple colour in the sodium nitroprusside layer indicates the presence of carbon disulphide, and the test made in this way will give a distinct reaction with 0·03 per cent. The purple fades quickly when the carbon disulphide is only present in small quantities, but can generally be reproduced, after standing, by shaking again. A considerable number of experiments was made to determine the best proportions in which to mix the reagents in this test, and it was found that the delicacy of the test was especially affected by the amount and the strength of the ammonia used. In some cases a green colour develops on standing, and this appears to be due to the sodium nitroprusside solution not having been prepared recently enough. It does not seem to be due to any impurities in the carbon tetrachloride. In this laboratory this test has proved more satisfactory than the xanthate test (Lunge, *Technical Methods*, III., 327) or the ammonium thiocyanate test.

IV. For Sulphur Chloride, etc.—A small silver coin (a two anna bit), preferably much worn, is cleaned with emery powder and soap and water, and allowed to lie in the carbon tetrachloride to be tested. In the presence of as little as 0·001 per cent. of sulphur chloride, S_2Cl_2, a darkening of the silver begins to show after ten minutes.

V. For Aldehydes and other Oxidisable Substances.—One c.c. of the carbon tetrachloride is shaken at intervals during one hour with 10 c.c. of a solution containing $N/1000$ potassium permanganate solution and $N/2$ sulphuric acid. The permanganate should not be entirely decolorised. The test can be made quantitative by running a blank and titrating the residual permanganate with $N/1000$ oxalic acid. Schiff's test for aldehydes was also used, the carbon tetrachloride being shaken with the reagent; this will detect 1 part of benzaldehyde in 10,000

VI. For other Organic Substances.—The carbon tetrachloride, when shaken with strong sulphuric acid, should not colour the acid.

The above tests will detect, in the proportions noted, any impurities which seem likely to occur, and good specimens of carbon tetrachloride will pass them all. It is suggested that such a quality be demanded of any specimen of this drug to be used medicinally.

Clive Newcomb.

The Chemical Examiner's Laboratory,
Madras.

NOTE ON THE DENIGÈS TEST FOR BUTYRIC ACID.

A method for the detection and estimation of butyric acid is given in an abstract in the Analyst (1918, **43**, 145). A pink colour is developed on treating a dilute solution of butyric acid with hydrogen peroxide in presence of ferrous iron, and, after the removal of the iron, adding sodium nitroprusside and acidifying.

The process has been investigated with a view to its application to the detection and estimation of small quantities of butter in margarine.

It was first noted that the quantity of hydrogen peroxide recommended in the case of the more dilute solutions was insufficient to oxidise the iron, and that the amount of sodium hydroxide mentioned was indefinite; but conditions were found for the reaction to proceed, and the colour, varying in intensity with the amount of butyric acid present, was regularly obtained.

To determine whether the test was specific for butyric acid, caproic acid was substituted. The colour was again produced, leading to the supposition that the caproic acid (b.pt., 201° to 203°) was contaminated.

Carefully fractionated caprylic acid (b.pt., 234·5° at 754 mm.) reacted similarly, however, the colour intensity indicating a degree of contamination with butyric acid equal to about 20 per cent.

A fraction of higher boiling point was next tried with similar results. Attempts were then made to remove any possible traces of butyric acid from the higher homologue (b.pt., higher than 234° C.) as follows:

(*a*) Repeated washing with hot water in a separating funnel.
(*b*) Fractional salting out of the sodium salts.
(*c*) Recrystallisation of the acid from 500 times its weight of water.

By none of these methods was it possible to detect any diminution in the proportion of butyric acid as indicated by the test.

The Reichert distillate from coconut oil also gave a positive reaction, although this oil is stated to be free from butyrin.

Acetic acid yielded no colour.

It appears to be certain that the fatty acids higher in the series than butyric give a similar reaction, and that the test fails as a method of estimating small quantities of butyric acid.

The writer is indebted for the suggestion of the problem to Mr. G. D. Elsdon, in whose laboratory the tests were carried out.

F. BAMFORD.

THE MUNICIPAL LABORATORY,
SALFORD.

THE ESTIMATION OF CHROMIUM.

ALTHOUGH the results given by Mr. Britton in his paper on the estimation of chromium (ANALYST, 1924, 130), go further to prove the accuracy of the alkaline oxidation method, he is mistaken when he says that "Up to the present no one appears to have used sodium peroxide, as such, in the volumetric estimation of chromium."

As a matter of fact, the method has been in common use, at least by leather trade chemists, for the estimation of chromium in chrome tanning liquors, since (I believe) the early days of chrome tanning, and is described in most text books on leather chemistry.

It appears in Prof. Procter's well known Pocket Book, and also in the present writer's *Practical Leather Chemistry*. It has always been considered an accurate method, but iron should be removed by filtration from the alkaline solution of chromate (*cf. Practical Leather Chemistry*, p. 87).

A. HARVEY.

196, BERMONDSEY STREET,
LONDON, S.E.1.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

SAND IN CINNAMON.

On March 24th a tradesman was summoned at Thornaby-on-Tees for the sale of cinnamon adulterated with sand. According to the analyst's certificate the sample contained 3·1 per cent. of silicious matter, instead of only 2·1 per cent., and the additional matter was practically all sand.

For the defence it was urged that, as cinnamon grew in sandy soil, it was practically impossible to keep it free from sand. Moreover, no standard for the permissible amount of silicious matter in cinnamon had been fixed by the British Pharmacopœia, since it was not possible to do so. Exception was also taken to the certificate on the ground that it did not state the component parts of the sample.

The Bench held that the certificate was not adequate and dismissed the case.

CHESHIRE CHEESE.

On March 14th a grocer was summoned at Salford for selling Dutch cheese in place of Cheshire cheese, which was demanded by the purchaser. Evidence was given that the cheese was sold at 1s. 2d. per lb., whereas the price of Cheshire cheese at that time was about 1s. 6d. per lb.

On analysis the sample was found to be deficient in fat to the extent of 26 per cent., and the Public Analyst stated in his certificate that "A genuine Cheshire cheese never contains less than 45 per cent. of fat calculated on the water-free cheese."

Mr. J. O. Garner, Secretary of the National Farmers' Union, stated that Cheshire cheese should be a full cream cheese, containing 28 to 30 per cent. of fat, which would correspond to 47 to 50 per cent. on a dry basis. Notwithstanding the fact that between 40,000 and 50,000 cows had been slaughtered in Cheshire, the competition of the Dutch cheese had caused the demand for Cheshire cheese to be slow.

The defence was that instructions had been given to the assistant not to sell the cheese as Cheshire cheese, that although it was Dutch cheese it looked like Cheshire cheese, and that the sale had been made through carelessness.

The Stipendiary fined the defendant 40s.

FISH MEAL WITH EXCESSIVE OIL AND DEFICIENT PHOSPHATE.

A case was heard before the Justices at the Bromley Police Court on March 24th, 1924, when the Kent County Council prosecuted the sellers of a meal known as "Drikod" Fish Meal, under Section 6, Sub-Section (a) & (b) of the Fertilisers and Feeding Stuffs Act of 1906.

Defendants in this instance had sold their fish meal with a warranty which had combined the albuminoids and oil, and had therefore failed to give the respective percentages of oil and albuminoids, as required by Section 1 (2) of the Act. Also, whereas 15 per cent. of phosphates had been guaranteed, the fish meal was found to contain only 11·57 per cent.

The offence was admitted by the representative of the defendants.

The Official Agricultural Analyst for the County of Kent, Mr. F. W. F. Arnaud, F.I.C., in his evidence, stated that he had found this meal to contain 15·75 per cent. of oil, 32·75 per cent. of albuminoids, and 11·57 per cent. of phosphates. In his opinion this amount of oil was excessive, and it was important that the oil in fish meal should be low, because it had been ascertained that fish oil was liable to produce a taint in the flesh of an animal to which it was given. Also, as fish meal was given largely for its albuminoid, and partly for its phosphate contents, it was essential that the percentage of albuminoids should be as high as possible. The Association of Fish Meal, Fish Guano and Fish Oil Manufacturers, comprising nearly all the manufacturers of fish meal in Great Britain, had agreed that a good fish meal should contain not less than 55 per cent. of albuminoids, not more than 5 per cent. of oil, and about 16 per cent. of phosphates. As a result of his experience he had found that fish meals of good quality approximately conformed to this composition, and that this was the quality of fish meal required for feeding purposes. The sellers of this particular fish meal had given a combined guarantee of albuminoids and oil of 50 to 55 per cent., and the real composition of the meal was, therefore, not apparent to the purchaser. The composition of this "Drikod" Fish Meal corresponded with the product known as fish guano, which was sold only as a fertiliser, and guanos contained fish and fish wastes which did not enter into the composition of meals used for feeding purposes. Fish guano was of much less value commercially than fish meal.

The Bench, after considering the evidence, fined the defendants £10 for the offence under Section 6 (a) and £5 under Section 6 (b), and allowed the prosecution £6 6s. costs. At the same time they did not wish to state that the defendants were fraudulent.

SPIRITS SOLD WITH NOTICE OF DILUTION.

On March 12th, a publican was summoned at the Hampstead Police Court for selling rum containing 3½ per cent. of water in excess of that in a rum of 35 per cent. under proof. A similar summons was brought against another Hampstead licensee, for the sale of whisky 38·8 per cent. under proof.

In neither case was there any dispute as to the strength of the spirits sold, but it was urged for the defence that a notice was exhibited in the bar of each of the defendants to the following effect:—"All spirits sold in this establishment are of the same superior quality as heretofore, but, as required by the Food and Drugs Adulteration Act, they are now sold as diluted spirits, no alcoholic strength guaranteed."

The Bench were informed that, whichever way their decision went, it was intended to make an appeal to the High Court, and it was accordingly decided that, pending the result of the appeal, only the first case should be heard.

After hearing the arguments on each side, the Bench decided to convict the defendant on the ground that the notice was extremely ambiguous and misleading, and that it did not convey to the mind of the purchaser the fact that when he asked for rum he was to be supplied with spirits diluted in strength below the statutory limit. The sale was, therefore, prejudicial to the purchaser. As they did not consider the case a bad one, they fined the defendant 20s., with £5 5s. costs.

Notice of appeal was given and the Bench agreed to state a case.

Department of Scientific and Industrial Research.

FUEL RESEARCH BOARD.

METHODS OF ANALYSIS OF COAL. ESTIMATION OF NITROGEN.

EXPERIMENTS made subsequent to the issue of the description of the methods of analysis recommended by the Sampling and Analysis of Coal Committee of the Fuel Research Board (*cf.* ANALYST, 1924, 36), have shown that the particular modification of the Kjeldahl method for the estimation of nitrogen, described on pages 7 and 8 of their interim report[*], gives perfectly reliable results; there is a close agreement between the results of this method and those obtained by a proved modification of the Dumas method.[†]

It has also recently been proved that copper sulphate is *not* a suitable substitute for mercury, as stated in a footnote on page 7 of the interim report, since its use yields, in general, low results.

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[*] Fuel Research Board, Physical and Chemical Survey of the National Coal Resources, No. 2, "Interim Report on Methods of Analysis of Coal," 1923. Published by H.M. Stationery Office. Price 1s. 6d. net.

[†] "The Estimation of Nitrogen in Coal," by A. Baranov and R. A. Mott, *Fuel*, 1924, 3, 31, 49.

Collection of Legislative Prescriptions Concerning Cheese.[*]

UNITED STATES OF AMERICA.—Cheese must not contain less than 50 per cent. of milk fat.[†] Skim and part-skim milk cheese must be so marked, and in the case of Edam, Roquefort and Camembert cheese quantity of contents of packages must be stated.

ARGENTINE.—Addition of salicylic, boric acid and its salts is prohibited.

AUSTRALIA.—All export cheese must be graded superfine, 1st grade, 2nd grade or 3rd grade, and no foreign matter, except rennet, salt or colouring matter deemed harmless by the Minister, may be added.

AUSTRIA.—Margarine cheese must contain 5 per cent. of sesame oil.

Cream cheese must not contain less than 50 per cent. of fat in the dry substance.
Whole milk cheese not less than 40 " " " "
¾ " " " " " " 30 " " " "
½ " " " " " " 20 " " " "
¼ " " " " " " 10 " " " "
Skim milk cheese " " " 10 " " " "

Ratio of fat to protein on the dry substance must be about 1:0·83 for whole milk cheese, and 1:2 for ½ whole milk cheese. Gervais cheese must not contain less than 50 per cent. of fat, Camembert 30 per cent. and Camembert (fat) 40 per cent., and in each case the water percentage must be 62.

BELGIUM.—No mineral substances, other than salt, and no antiseptics may be used.

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[*] Summarised from the book published by Dr. A. J. Swaving, The Hague. (*Cf.* ANALYST, 1924, 207.)

[†] All Fat percentages are on the dry material.

BRAZIL.—

Cream cheese must contain 45 per cent. of milk fat in the dry substance.
Whole milk cheese must contain 35 „ „ „ „ „
½ fat cheese must contain 25 „ „ „ „ „
Skim milk cheese less than 25 „ „ „ „ „

CANADA.—Cheese must not contain less than 45 per cent. of milk fat in the dry substance, and cheese for export must be graded for flavour, texture, closeness, colour, and finish.

DENMARK.—(A) Hard Cheese.	Minimum Fat percentage content. (stamped on cheese).	Maximum Water percentage content.
Class I.	45	50
„ II.	30	54
„ III.	20	57
„ IV.	10	59
„ V. Skim milk	—	60
(B) Soft Cheese	(stamped on wrapper).	
„ VI.	45	60
„ VII.	30	60
„ VIII.	20	60
Roquefort	50	52
Camembert	45	60
Gervais	50	60
Guarg	—	65

Emmenthaler Cheddar must be in Class I., Danish Swiss in Classes I. or II., Gouda, Edam, Tilsit, Steppecheese in Classes I., II. or III., and factory cheese in classes III., IV. or V.

(*Note.*—These divisions are likely to be revised in the near future.)

ESTHONIA.—

Cream cheese must not contain less than 40 per cent. of fat in the dry substance.
Whole milk cheese „ „ „ „ 30 „ „ „ „
¾ fat „ „ „ „ „ 25 „ „ „ „
½ fat „ „ „ „ „ 20 „ „ „ „
¼ fat „ „ „ „ „ 10 „ „ „ „
Skim milk cheese less than 10 „ „ „ „

FINLAND.—Dairy factories have agreed that

Whole milk cheese should contain 45 per cent. of fat.
½ fat cheese „ „ 30 „ „ „ „
Skim milk cheese „ „ 15 „ „ „ „

FRANCE.—Camembert cheese must contain 36 per cent. of fat by weight on the dry substance, and be exclusively made from cows' milk. Further regulations for cheese in general are probable.

GERMANY.—"Proposals for Regulations concerning Foodstuffs, 1913," suggest minimum fat percentages for cream cheese, 50; whole milk cheese, 40; ¾ fat cheese, 30; ½ fat cheese, 20; ¼ fat cheese, 10; skim milk cheese, less than 10 in the dry substance. Modifications may be expected.

IRELAND.—Irish Free State manufacturers have agreed to 45 per cent. fat for whole milk cheese and 25 per cent. for skim milk cheese in the dry substance.

ITALY.—Cheese is considered to be made from margarine if the refractive index of the fat is above 48, and the Reichert-Meissl-Wollny number below 18.

If the latter is between 18 and 24 the cheese is considered suspicious, and if above 24 as genuine. Margarine cheese may not contain added colouring matter.

NETHERLANDS.—Manufacture of cheese is under strict Government control, and whole milk cheese must contain 45 per cent. of fat in the dry substance, and be stamped accordingly. Cheese made from partially skimmed milk must be stamped 40+, 30+, 20+, according to the proportion of fat contained.

NORWAY.—Cheese must be in one of the four following groups and be marked according to the fat content.

I. Whole milk cheese with not less than 45 per cent. of fat in the dry substance.
II. ½ fat ,, ,, ,, ,, ,, ,, 28 ,, ,, ,, ,,
III. ¼ fat ,, ,, ,, ,, ,, ,, 18 ,, ,, ,, ,,
IV. Skim milk cheese with less than 18 ,, ,, ,, ,,

It is likely that the limits for groups II. and III. will be raised.

POLAND.—Whole milk cheese must contain 30–40 per cent. of fat.
½ fat cheese ,, ,, 20–30 ,, ,, ,, ,,
Skim milk cheese ,, ,, 10–20 ,, ,, ,, ,,
and the maximum water content is 50 per cent. in the dry substance.

ROUMANIA.—Cheese from part skimmed cows' milk must not contain more than 75 per cent. of water, and sheep cheese more than 55 per cent., whilst mixed cheese from these milks must not have more than 70 per cent. of water. No other materials than lactic acid, rennet, salt, ferment or seasoning may be used in the manufacture of cheese.

SWITZERLAND.—
Whole milk cheese must contain not less than 45 per cent. of fat in the dry [substance.
¾ fat cheese ,, ,, ,, ,, ,, 35 ,, ,, ,, ,,
½ fat ,, ,, ,, ,, ,, ,, 25 ,, ,, ,, ,,
¼ fat ,, ,, ,, ,, ,, ,, 15 ,, ,, ,, ,,
Skim milk cheese less than 15 ,, ,, ,, ,,

The water content in Emmenthaler whole milk cheese must be between 26 and 36 per cent., and for ¾ fat Emmenthaler between 27 and 38 per cent.

STRAITS SETTLEMENTS.—
Whole milk cheese must not contain less than 50 per cent. of milk fat in the dry [substance.
Skim milk cheese ,, ,, ,, ,, ,, 10 ,, ,, ,, ,, ,,
Cream cheese ,, ,, ,, ,, ,, 60 ,, ,, ,, ,, ,,

No foreign fat, preservative other than salt, or colouring matter other than harmless vegetable colouring matter may be added.

SOUTH AFRICA.—Cheese must be graded into one of 3 grades for export according to flavour and aroma, quality (including body and texture), colour, salting, finish and general appearance.

NEW ZEALAND.—Quality of export cheese is rigorously controlled by Government, and standardised grading is being aimed at.

VENEZUELA.—Starch and gelatin may be added if stated, and all cheese not made with cows' milk must be marked in accordance with its origin, and the rind of artificial cheese must be coloured red. No cheese containing cheese-mites may be sold.

No special standards are in force in the following countries:—Algeria, British India, Bulgaria, Chili, Czecho-Slovakia, Grand Duchy of Luxemburg, Great Britain, Greece, Hungary, Lithuania, Livonia, Portugal, Serbia, Sweden, or Tunis.

D. G. H.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Detection of Nitrates in Milk. M. E. Pozzi-Escot. (*Bull. Soc. Chim.*, 1924, **35**, 72).—The detection of nitrates in milk by means of brucine or diphenylamine necessitates the use of concentrated sulphuric acid, which acts on the proteins and lactose, and is prone to produce colorations similar to that given by a trace of nitrate. This inconvenience may be avoided by precipitating the casein and fatty matter by heating the milk with sulphuric acid, neutralising the whey with ammonia and evaporating it to a syrup or even to dryness, and triturating the residue with cold, slightly diluted sulphuric acid and a little ether. The ether, which will contain the nitric acid of any nitrate present in the milk, is decanted, neutralised with a drop of ammonia solution, and evaporated at a low temperature on the water-bath, being the residue tested for nitric acid by any of the ordinary methods. T. H. P.

Composition of Canned Salmon. O. E. Shostrom, R. W. Clough and E. D. Clark. (*Ind. Eng. Chem.*, 1924, **16**, 283–289.)—The following table gives a summary of the results obtained in an investigation of the variations in the composition of salmon and trout as influenced by species and locality where caught. In the case of the special samples, the tins examined contained approximately the same section of an individual fish; with the exception of the bone content, all the results are calculated on the bone-free substance.

Species of Fish.	Number of Samples.	Bone Per Cent.	Moisture Per Cent.	Fat Per Cent.	Salt-free Ash Per Cent.	Proteins Per Cent.
Special samples.						
Sockeye salmon	126	1·96	67·19	8·58	1·32	21·04
Chinook salmon	216	1·78	63·98	13·51	1·21	19·51
Coho salmon	99	1·88	67·49	8·49	1·24	21·08
Pink salmon	90	2·32	70·05	6·20	1·31	20·56
Chum salmon	108	1·87	70·85	5·15	1·28	21·48
Steelhead trout	20	1·67	57·70	20·09	1·21	19·95
Commercial samples.						
Sockeye salmon	41	2·37	64·79	11·22	1·23	20·80
Chinook salmon	24	2·21	63·17	15·72	1·21	17·67
Pink salmon	9	2·53	69·80	6·99	0·76	21·40
Chum salmon	33	2·33	66·95	10·52	1·02	20·67
Steelhead trout	14	2·40	66·84	8·95	1·21	21·32
Atlantic salmon	6	1·14	64·30	12·49	1·22	21·14

The fat content of different salmon of the same species caught in the same locality varied considerably (from 6·7 to 27·2 per cent.); it also varied greatly between different species, localities, and years, the extreme variation being from 2·65 to 27·26 per cent. W. P. S.

Nature of Corrosion in Canned Fruits. E. F. Kohman and N. H. Sanborn. (*Ind. Eng. Chem.*, 1924, **16**, 290–295.)—The acidity of fruits is not the chief cause of perforations in the tin containers or of hydrogen formation ("blown" tins), since there is no relation between the hydrogen ion concentration of the different fruits and the extent to which they cause trouble. The fact that perforations, etc., are more common in varnished tins than in plain tins indicates that the substance causing the trouble is present in small quantity; its action is concentrated on minute unprotected parts of the varnished tins, but is less noticeable on the entire surface of the plain tins. A small amount of oxygen acts in this way in the case of canned apples, but the removal of oxygen by reduced pressure does not entirely eliminate perforations with all fruits, as it does with apples. Some fruits appear to contain oxidising substances other than oxygen, and it is suggested that these substances may be intermediary respiratory substances (oxyhaemoglobin, or possibly the fruit colours, the anthocyans). The fact that oxygen disappears slowly and hydrogen formation follows soon after its disappearance in varnished tins, whilst in plain tins the oxygen disappears rapidly, but hydrogen formation is delayed, indicates that there is a protection of some kind in the tin against acid alone, and that the combination of acid and oxygen is able to break down this protection and allow the acid to continue its action even after the oxygen has been used. There is no evidence as to the nature of this protection. W. P. S.

Apparatus for the Estimation of Tin in Canned Foods. E. J. B. Willey. (*J. Soc. Chem. Ind.*, 1924, **43**, 70–72 T.)—The ordinary gravimetric estimation of tin in foods suffers from two disadvantages; one, that it is needful to take a large quantity of the sample for the initial wet combustion, and two, that unless the tin is dissolved and re-precipitated it is liable to be contaminated, especially with silica, and so yield high results. The volumetric method as ordinarily employed tends to give low results on account of atmospheric oxidation. An apparatus is described and figured which enables the reduction and titration to be conducted entirely in an atmosphere of carbon dioxide. With the use of this method a smaller quantity of the sample (25 grms.) may be taken for the initial combustion, which is carried out as usual; the tin is precipitated as sulphide, filtered on asbestos, and dissolved in dilute hydrochloric acid with the aid of potassium chlorate. The solution is then boiled to expel chlorine and introduced into the apparatus, reduced by means of zinc foil, and finally titrated with 0·005 *N* iodine solution. H. E. C.

Estimation of Starch and Sugars by the Use of Picric Acid. M. R. Coe and G. L. Bidwell. (*J. Ass. Off. Agric. Chem.*, 1924, 7, 297–304.)—The picric acid colorimetric method hitherto used in biological work (ANALYST, 1921, 288, 508) may be applied to the estimation of starch or sugar in feeding stuffs and similar materials. In the case of starch, the preliminary hydrolysis cannot be effected by means of taka-diastase, since concordant results are unobtainable in that way; if, however, a cold water extract of malt is used, the results are satisfactory.

When reducing sugars are to be estimated, the colour is developed immediately the sodium carbonate is added to the solution. With sugars that require hydrolysis, however, advantage is taken of the hydrolysing properties of the picric acid, and the alkali is added only after the hydrolysis has taken place.

When the starch or sugar is accompanied by mucilaginous substances such as are found in linseed cake, apple pomace, or pectin pulp, or by polyphenols, aldehydes, ketones, purine bases, the colouring matters present in molasses and the like, this colorimetric method yields high results; such disturbing substances may, however, often be eliminated by special preliminary treatment.

The procedure to be followed in the case of starch and in that of sugars is given in detail. The advantages of the method are its applicability to small samples or to materials containing small proportions of starch or sugar, the saving of time in comparison with the copper reduction method, and the simplicity of the manipulation, preparation of crucibles and the handling of bulky solutions being avoided. T. H. P.

Detection of Fruit Wines (Cider or Perry) in Ordinary Wine. F. Schaffer. (*Mitt. Lebensm. Unters. Hyg.*, 1923, 15; *Ann. Chim. anal.*, 1924, 6, 88). —The author replies to Widmer's objections to his method for detecting cider in wine, this being based on the diminished reducing power of the wine. The following modified procedure is now recommended:—Forty c.c. of the wine are decolorised by boiling with 3 grms. of pure animal charcoal and filtered, and the filtrate is heated to boiling with excess of precipitated calcium carbonate. After further filtration the cooled liquid is treated, slowly and with shaking, with 8 c.c. of 10 per cent. barium acetate solution, and again filtered. Five c.c. of the neutral filtrate are mixed with 2 drops of 0·2 *N* silver nitrate solution, and, after being made alkaline by addition of 1 c.c. of 0·1 *N* sodium hydroxide solution, are placed in the dark, and the time elapsing before distinct reduction is apparent is noted; the test should be made in duplicate or even in triplicate. With cider the reduction becomes evident in a few minutes, and, with wines containing a marked proportion of cider, in 10 to 20 minutes; in some cases comparison of the suspected wine with the natural wine of known purity is advisable. T. H. P.

Evaluation of Charcoals used for the Decolorisation of Tinted White Wines. X. Rocques. (*Ann. Chim. anal.*, 1924, 6, 65–72.)—No matter what care is taken to separate the juice rapidly from the stalks prior to fermentation, white wines prepared from grapes with colourless juice but coloured skin often exhibit a slight colour. In France it is permissible to decolorise such wines by means of animal black purified by treatment with hydrochloric acid to dissolve the alkaline earth carbonates and phosphates, and then freed from the excess of acid by washing with water. For the examination of such decolorising carbons, which are supplied mostly as paste (since they lose in value when dried) the following methods are recommended:—The moisture, total ash, and ash insoluble in hydrochloric acid are estimated on 5 grms. of the material. There being no definite correlation between the decolorising power and moisture content, no maximum value to be allowed for the latter is suggested. The ash soluble in hydrochloric acid should be small in amount, a maximum of 1 per cent., calculated on the material as supplied, being suggested.

Reaction and chlorides:—Ten grms. of the black are shaken with 100 c.c. of distilled water and, after standing for 24 hours, filtered off. The reaction of the filtrate should not be acid towards litmus in comparison with that shown by the original water; further, the filtrate should contain not more than 0·1 per cent. of chlorides calculated as sodium chloride. When 5 grms. of the black are shaken for 15 minutes with 200 c.c. of a 10 per cent. aqueous solution of tartaric acid, and the liquid is filtered, the acidity, estimated by titration with 0·05 N sodium hydroxide solution and corrected for the slight dilution involved, should not be diminished by more than 7 or 8 per cent.; moreover, the tartaric acid solution should not dissolve more than 0·04 grm. of iron per 100 grms. of the black.

The decolorising power of the charcoal is tested as follows:—A pink liquid is prepared from red wine and 10 per cent. tartaric acid solution, to match a solution containing 8 c.c. of 0·1 N potassium permanganate solution, and 16·8 c.c. of 0·1 N potassium dichromate solution per litre. The amount of the black just required to decolorise 200 c.c. of the pink wine when shaken therewith in a half-litre bottle for 15 minutes is then determined. With paste black of good quality 200 grms. are required per hectolitre of the wine. T. H. P.

Chemical Composition of Sesame Oil. G. S. Jamieson and W. F. Baughman. (*J. Amer. Chem. Soc.*, 1924, **46**, 775–778.)—The oil expressed from Chinese yellow sesame seeds has been examined by well-known methods. The saturated and unsaturated acids were separated, and the former were methylated and separated by fractional distillation under reduced pressure, and the usual constants determined on the fractions, from which the fatty acids were subsequently liberated and crystallised. The unsaturated acids yielded no hexabromide, but linolic tetrabromide separated, and calculation from the iodine value showed the presence of oleic acid. The percentage composition of the glycerides is calculated to be as follows:—Oleic, 48·1; linolic, 36·8; palmitic, 7·7; stearic, 4·6; arachidic, 4; lignoceric acid, trace; and unsaponifiable matter, 1·7.

H. E. C.

Comparative Analytical Study of Various Oils in the Chaulmoogra Group. G. A. Perkins. (*Philippine J. Sci.*, 1923, **23**, 543–569.)—The oils from 9 species of authenticated seeds related to Chaulmoogra (*Gynocardia odorata*; *Hydnocarpus alcalae*; *H. anthelmintica*; *H. Hutchinsonii*; *H. subfalcata*; *H. venenata*; *H. Wightiana*; *H. Woodii*; *Pangium edule*) and 15 commercial samples of *Taraktogenos kurzii* oil were examined. Fractional distillation of the ethyl esters of 13 of the oils and crystallisation of the recovered fatty acids from each of four fractions were carried out, and the eight resulting fatty acid fractions were examined for freezing point, iodine absorption, specific rotatory power and saponification values. The results obtained from the whole examination confirm the exclusion of *Gynocardia odorata* from the Chaulmoogra group. A close similarity between the oil of *Taraktogenos kurzii* and *Hydnocarpus* oils was established, and the only *Hydnocarpus* oil found to differ materially was that of *H. alcalae*, which contained a large proportion of chaulmoogric acid and little or no hydnocarpic acid. From a therapeutic point of view there appears to be no reason for considering Chaulmoogra oil superior to *Hydnocarpus* oils, but clinical confirmation of this point is needed. It appears doubtful whether *Pangium edule* oil contains either chaulmoogric or hydnocarpic acids, although some variable optically active constituent is present in the fatty acid fraction, possibly the above-mentioned acids, together with a destructive enzyme. Nine tables are given in the paper comprising (1) Physical and chemical data already available on Chaulmoogra and related oils, (2) Extraction of the oils, (3) Characteristics of the oils, (4), (5), (6), (7), (8), and (9) Characteristics of the fractions. D. G. H.

Detection of Adulteration of Cacao Butter. A. Koehler. (*Comptes rend.*, 1924, **178**, 940–941.)—Under similar conditions of temperature and of shaking, the volume of ethylacetoacetate which must be added to cacao butter to produce a standard turbidity is a constant dependent upon the purity of the sample, and hence is useful for estimating impurities or adulterants; it is more sensitive than the determination of the usual constants. An adulteration of 15 per cent. with other vegetable butters is detected with certainty. As the number of c.c. of the ester required to produce the standard turbidity is largely influenced by temperature it is best to titrate the sample and one of known purity, for comparison under identical conditions. The method is to run the ester from a burette into a 20 per cent. solution of the cacao butter in chloroform at a temperature of 15–20° C., until the appearance of a turbidity which is not increased by a further drop (an end point quite easily recognised). The number of c.c. of ester required by 2 c.c. of the 20 per cent. solution is called "the turbidity index," and is remarkably constant for pure cacao butters, but is increased by an adulterant. In the case of samples grossly adulterated the ester may precipitate the adulterant; in such cases the solution is diluted with a 20 per cent. solution of a pure cacao butter. H. E. C.

Brucine in Strychnine Nitrate. Reck. (*Chem. Zeit.*, 1924, **48**, 166.)—Strychnine nitrate not infrequently contains brucine. The following modifications of Keller's method is used by Gordin for the quantitative separation of strychnine and brucine:—A quantity of 0·2 grm. of the alkaloid is dissolved in 15 c.c. of 3 per cent. sulphuric acid, and, when cold, treated with 3 c.c. of a cold mixture of equal parts of nitric acid (1·42) and water. Brucine is decomposed, with the formation of oxidation products, which are not soluble in chloroform made alkaline with sodium hydroxide solution. Therefore the mixture is shaken in a separating funnel in the presence of excess of sodium hydroxide solution with successive quantities of chloroform (20 c.c., 10 c.c., and 10 c.c.). The chloroform extracts are evaporated, and the residue of strychnine dried at 110° C. until the weight is constant. A freshly-prepared mixture of 0·1002 grm. of strychnine nitrate and 0·1021 grm. of brucine nitrate gave, on analysis, 0·1004 grm.

of strychnine nitrate. Results of investigations of 6 different commercial samples of strychnine nitrate containing brucine are given. The advisability is discussed of calling the preparations which contain more than 50 per cent. of brucine nitrate, "brucine nitrate containing strychnine," since brucine is very inactive compared with strychnine. P. H. P.

Microcrystalline Reaction for Identifying Traces of Cantharidin. G. Denigès. (*Bull. Soc. Pharm. Bordeaux*, 1923, **61**, 63; *J. Pharm. Chim.*, 1924, vii, **29**, 106.)—As little as 0·1 or 0·05 mgrm. of cantharidin may be identified by adding one or two drops of chloroform to the material on a slide and allowing the solvent to evaporate; the cantharidin is left in tablets or prisms showing characteristic step-like incisions. The sublimed product also exhibits distinctive form. In either case, addition of a drop of benzene to the crystals renders these more distinct. Even 0·001 mgrm. of cantharidin may be detected if it is concentrated in as little solvent as possible before crystallisation. T. H. P.

Biochemical, Bacteriological, etc.

Preservation of Blood by means of Formaldehyde. J. C. Bock. (*J. Biol. Chem.*, 1924, **59**, 73–76.)—It does not seem advisable to use the average formaldehyde as a preservative for specimens of blood intended for blood sugar estimation by the revised Folin-Wu method (*J. Biol. Chem.*, 1920, **41**, 367) as recommended by Denis and Aldrich (*J. Biol. Chem.*, 1920, **44**, 203). They claim that formaldehyde in certain quantities will affect neither the alkaline copper tartrate solution used nor the tungstic acid filtrate. The author describes experiments in which he used six different samples of formaldehyde, and gives a table of results showing the effect of adding varying amounts of the different formaldehydes to glucose solution, tungstic acid filtrate and blood specimens before precipitation. Five out of six samples of the formaldehydes investigated reacted with the alkaline copper tartrate, thereby materially influencing the results. In several cases there was an increase in the amount of reduction, expressed in terms of glucose, of more than 40 per cent. Denis and Aldrich must have investigated an inadequate number of formaldehyde samples. P. H. P.

Enzyme Actions of Beef Tissues. K. G. Falk, H. M. Noyes and K. Sugiura. (*J. Biol. Chem.*, 1924, **59**, 213–223.)—The ester-hydrolysing actions of six beef tissues, brain, kidney, spleen, liver, heart-muscle, and lung were determined on ten purified esters, *viz.* methyl acetate, ethyl acetate, isobutyl acetate, phenyl acetate, benzyl acetate, glyceryl triacetate, methyl butyrate, ethyl butyrate, methyl benzoate and ethyl benzoate, and the protease actions on two protein preparations, peptone and casein (0·1 grm. of each of the latter in each test). The beef (steer) tissues were ground and extracted with water, and toluene was added and was present throughout the tests. The mixtures were kept in an ice-box overnight and filtered through paper the next day. Portions of 5 c.c. each of the filtrates were diluted with water to 15 c.c. and tested, and the residues from the extraction were also tested. The degree of lipase actions was determined by titration with 0·1 *N* sodium hydroxide solution, with phenolphthalein as indicator. For the protease actions the formaldehyde method (with phenolphthalein and 0·1 *N* sodium hydroxide solution) was used, each test being made in duplicate. The temperature was maintained at 38° C. for 22 hours, and the solutions were all brought to an initial P_H value of 7·0. The results are presented in the form of tables for the absolute actions and curves for the relative actions. The characteristic relations for the various tissues are discussed and compared with those for the corresponding rat tissues. It is possible to plot the relative actions caused by each tissue on a number of different substrates so that a characteristic "picture" of the enzyme action is obtained, as can be done for rat tissues. The "pictures" for the actions of beef kidney, liver and lung are quite characteristic. The three other beef tissues show "pictures" more or less similar to the Flexner-Jobling rat carcinoma "picture." Explanations for similarities and differences will be advanced when data for other animal tissues have been gathered. P. H. P.

Evaluation of Saponins as Frothing Agents. L. Kofler. (*Chem. Zeit.*, 1924, **48**, 165–166.)—The determination of the toxicity of saponins by haemolysis action for deciding whether they should be used as frothing agents in foods is one-sided and often leads to false suppositions. Not many data concerning the frothing power are known. The author gives the following table:

Saponin.	Hæmolytic Index.	Frothing-Number	$\frac{\text{Poison}}{\text{Froth}}$-Quotient.
Guaiacum-Saponin, Merck.	660	28,500	0·023
Sapotoxin. Merck.	30,000	20,000	1·5
Sapindus-Saponin. Hoffmann La Roche	28,600	16,700	1·7
Aphrogen	2,000	800	2·5
Horse-chestnut-Saponin, Merck. ..	10,000	3,300	3·0
Saponin pur. albiss., Merck.	25,000	2,500	10·0

He finds that the haemolytic action of aphrogen towards red blood corpuscles and white mice is smaller than that of other saponins. This confirms the work of Mandelbaums. On the other hand, the frothing power is smaller than that of other commercial saponins, *e.g.* 25 times smaller than that of sapotoxin and 36 times smaller than that of guaiacum-saponin. The frothing number was determined according to the author's method (ANALYST, 1922, **47**, 403). Experimental conditions for a froth one cm. high were found. By comparing the haemolytic action with the frothing power, *i.e.* by dividing the haemolytic index by the frothing number, the (poison: froth) quotient is obtained which without further data gives the usefulness of the saponin as a frothing agent for foods. The smaller the haemolytic index and the greater the frothing number, the smaller is the poison: froth-quotient. By comparing the quotient of aphrogen with that of sapotoxin it is found that, on making lemonades with aphrogen and sapotoxin and adding so much of each saponin that both drinks have the same frothing power, the haemolytic action of the aphrogen-lemonade is about 67 per cent. greater than that of the sapotoxin-lemonade.

Details are given of subcutaneous injections on white mice. The fatal dose for a medium-sized mouse is 0·03 grm. of aphrogen, but 0·00075 grm. of sapotoxin; Therefore, judging by this, aphrogen is 40 times less poisonous than sapotoxin, but since, according to the table, the frothing power of aphrogen is 25 times less than that of sapotoxin, so, from injection experiments, the toxicity of a sapotoxin-lemonade is about 60 per cent. greater than that of an aphrogen-lemonade, when both froth equally. Saponins can only be evaluated as frothing agents by comparing the physiological action with the frothing number and obtaining the poison: froth quotient.

P. H. P.

Effect of Storage of Livers on the Vitamin *A* Potency of Cod Liver Oil. A. D. Holmes. (*Ind. Eng. Chem.*, 1924, **16**, 295–297.)—Cod liver oils were rendered from fresh cod livers, from livers which had been stored in ice out of contact with air for six months, and from livers stored for one year under similar conditions. Albino rats suffering from vitamin *A* starvation were fed with the three classes of oil, and the results obtained showed that the oils from the stored livers were as efficient as the oil from the fresh livers. One mgrm. per day of each of the oils contained sufficient vitamin *A* to promote good growth in the rats.

W. P. S.

Amount of Available Insulin in the Pancreas of Domestic Animals. F. Fenger and R. S. Wilson. (*J. Biol. Chem.*, 1924, **59**, 83–90.)—This investigation was carried out for the purpose of obtaining some definite information regarding the amount of insulin present in the pancreas of cattle, hogs and sheep. The method of separating the active substance, a modification of that of Doisy, Somogyi and Shaffer (*J. Biol. Chem.*, 1923, **55**, 31) is described in detail; also the method of standardising the purified insulin. The preparations were tested on rabbits, and a table of results is given. The insulin unit chosen by the authors is the amount of insulin per kilo. of body weight necessary to produce convulsions and coma within 5 hours in from 60 to 70 per cent. of rabbits weighing between 1 and 2 kilos. Larger rabbits respond as promptly to their quota of insulin as do the smaller ones, and the convulsive dose of insulin is in direct proportion to ths body weight within these weight ranges. The authors find that the amount of insulin in the pancreas of domestic animals varies from 1,500 to 2,200 rabbit units per kilo. of fresh glands. The average yield approximates 1,800 units from cattle, hogs and sheep. The domestic animals slaughtered yearly in the United States under Federal Government inspection furnish about 5½ million kilos. of fresh pancreas glands, so that the available supply of raw material is far in excess of any possible demand for insulin. Insulin solution may be sterilised with impunity. Absolutely fresh glands, still warm if possible, are necessary for insulin production, and a grinding process, in addition to the mincing, breaks up the cell walls and renders the active substance available for extraction. An experiment described shows that the presence of acid is required to bring insulin into solution in either water or alcohol. The samples have shown no decrease in strength over a period of several months. There seems to be no doubt that insulin is of a protein nature, probably a derived and not a native protein. The most active products at the authors' disposal possess the physical and chemical characteristics of complex protein derivatives not lower than the primary proteoses.

P. H. P.

Method of Measuring the Activity of Laccase. P. Fleury. (*Comptes rend.*, 1924, **178**, 814–816.)—A current of air is passed for a definite time through 10 c.c. of guaiacol solution mixed with the laccase to be tested, the solution being placed for the purpose in a special wash-bottle in a thermostat. The guaiaquinone thus formed is extracted by shaking the solution with 10 c.c. of chloroform, and is estimated colorimetrically. The results of a series of tests show that, within certain limits of time and of the concentration of the enzyme, laccase resembles diastase in obeying the law of proportionality between the amount of substrate transformed and both the time and the quantity of enzyme. Within such limits it is possible to obtain a measurement of the activity of a laccase preparation.

T. H. P.

Chemotherapeutic Experiments with Chaulmoogra and Allied Preparations. O. Schöbl. (*Philippine J. Sci.*, 1923, **23**, 533–542; 1924, **24**, 23–27).—I. *The Growth-Inhibiting Activity of Chaulmoogra Oil and its Derivatives toward Bacillus tuberculosis in vitro.*—This was tested by adding measured amounts of the oils to test tubes containing 10 c.c. of 5 per cent. glycerin agar, and it was found that chaulmoogra oil has considerable inhibiting power over *B. tuberculosis*, which is specific, *i.e.* it occurs in dilutions of the oil in which no inhibition of non-acid-fast bacilli can be discerned. Oils from plants related to *Taraktogenos kurzii* behave similarly. *Hydnocarpus Wightiana, H. alcalae, H. subfalcata,* and *H. venenata* (all containing optically active acids) are decreasingly active in the order given, whilst the oil from *Gynocardia odorata* (containing no optically active acids) is inactive. The sodium salts of chaulmoogra oil acids were found to vary in their activity, that prepared from isolated hydnocarpic acid approaching in activity the soap from the total fatty acids, whilst the sodium salt of chaulmoogric acid was far inferior in its growth-inhibiting effect.

II. *Comparison of the Antiseptic Power of Chaulmoogra Oil with that of other Vegetable and Animal Oils, rare and common.*—Although certain vegetable oils containing optically active fatty acids are found to inhibit the growth of acid-fast bacteria *in vitro*, they do not do so to such a high dilution as Chaulmoogra and Hydnocarpus oils, which contain the optically active fatty acids. A table is given showing the growth-inhibiting "titer" (*i.e.* the amount of oil which, when added to 10 c.c. of agar, still produced inhibition of growth) and growth-inhibiting values (*i.e.* the relative strength of the inhibiting effect) for 42 different oils. It may be noted that certain essential oils and oils containing volatile constituents show very high selective inhibitory action upon acid-fast bacilli. D. G. H.

Viability of Intestinal Pathogenic Bacteria in Fruits and Philippine Foods eaten Raw. A. Vasquez-Colet. (*Philippine J. Sci.*, 1924, **24**, 35–39.)—The foods used in the experiments were bagong (small shrimps pickled in salt), patis (obtained from bagong by extraction and boiling), both sold in the market and eaten raw, vinegar (obtained by fermentation from the Nipa palm), mango, banana, chico, lanzones, apples, quesco, and guava. It was found that the cholera vibrio will survive from a few hours to at least 6 days, but is less long lived when mixed with human faeces; typhoid bacillus survives from a few minutes to at least 3 days; and dysentery bacillus (Flexner and Shiga types) from a few minutes to at least 5 days. The results show that such articles of food eaten raw may convey infection, but not to a marked extent, and in the case of foods rich in bacteria, such as patis, or in the fermenting state, survival of the cholera vibrio is very limited in duration. D. G. H.

On the Cancer-Producing Factor in Tar. E. L. Kennaway. (*British Med. J.*, 1924 [March 29], 564–567.)—A summary is given of the evidence, from both industrial and experimental sources, which indicates the presence or absence of cancer-producing power in the different fractions obtained from coal tar and in the pure substances isolated from it. Of the different kinds of tar, those which produce cancer are lignite tar, gasworks tar, some forms of producer-gas tar, and probably coke-oven tar, whilst blast furnace tar does not. The chief differences between blast-furnace tar and the ordinary cancer-producing gasworks tar are that the former contains greater quantities of paraffins and of phenols other than carbolic acid, but is deficient in some aromatic compounds found in the latter. The industrial evidence alone shows that the cancer-producing substance in gasworks tar is present in the fractions of higher boiling point, *viz.* creosote oil, anthracene oil (and hence in the green oil and crude anthracene) and pitch; the experimental evidence derived from mice supplements this by showing that the substance is not concentrated in the solids suspended in anthracene oil, and that it is present in the distillate of highest b.pt. obtainable from pitch. Hence it may distil through an interval of temperature extending roughly from 250° C. (the creosote fraction) to above 500° C. (the "pitch distillate"). So far, attempts to find the cancer-producing substance among the known constituents of coal tar have given wholly negative results. Anthracene, phenanthrene, chrysene, picene, retene, truxene, acenaphthene, fluorene, acridine, carbazole, aniline benzene, toluene and xylene have been excluded by experimental tests (several of which are described in detail); and either the experimental or industrial evidence, or both of these, also excludes naphthalene, the acids, bases and other nitrogenous compounds, paraffins, olefines and naphthenes. It now seems not unlikely that the cancer-producing substance is a compound, as yet unknown, which is unstable and present in amounts as small as those of the vitamins in foods; as in the case of some hormones, its identification may be long delayed, even when very concentrated preparations can be obtained.

A long list of references to the chemical and medical literature of the subject is given.

Toxicological and Forensic.

Influence of Blood on Chemical Reactions. G. Beccadelli. (*Arch. Farm. Sper.*, 1923, 36, 137–144; *Chem. Abstracts*, 1924, 18, 406).—The presence of blood has a pronounced influence on the reduction of ammoniacal silver nitrate solution by formaldehyde, the colour of the colloidal silver varying with the species of animal from which the blood is derived. For the identification of blood stains on fabrics, about 5 sq. mm. of the material are cut out and macerated for 24 hours in 1 c.c. of water at the ordinary temperature. About 0·5 c.c. of the extract is transferred to a small test tube and treated with 0·5 c.c. each of 38 to 40 per cent. formaldehyde, 0·75 per cent. silver nitrate solution and ammonia solution of sp. gr. 0·925. The tube is inverted two or three times and allowed to stand. In the case of human blood a characteristic amber yellow coloration develops within 16 hours if the stain was not recent, or a turbidity if it was recent. Failure to obtain this coloration may indicate that the stain was old human blood or the blood of a lower animal. The colorations produced by the blood of different animals are as follows:—Rabbit, amber yellow, not very pronounced; goat, amber yellow, still less pronounced; dog, brick red; sow, straw yellow; ox, brass yellow. The differences in colour are attributed to differences in the size of the colloidal silver particles.

Water Analysis.

Soluble Aluminium and the Haematoxylin Test in Filtered Waters. W. D. Hatfield. (*Ind. Eng. Chem.*, 1924, 16, 233–234.)—In cases where water is treated with aluminium sulphate previous to filtration, the filtered water will, in all probability, contain traces of soluble aluminium if the P_H value of the water is less than 5·7 or more than 7·3, the minimum solubility of aluminium hydroxide in carbonate and bicarbonate solutions lying within these limits. The following method is proposed for the estimation of small quantities of aluminium (*e.g.* 0·1 part per million of water):—It consists in the formation of the haematoxylin-aluminium colour compound in a portion of the water which has been adjusted to P_H 8·2 to 8·3; the mixture is then acidified to P_H 4·5, in order to destroy the lavender coloration given by iron and other metals which may be present. Fifty c.c. of the water to be tested are treated in a Nessler cylinder with 1 c.c. of saturated ammonium carbonate solution (this adjusts the P_H value to 8·2) and 1 c.c. of 0·1 per cent. haematoxylin solution; after fifteen minutes, the mixture is acidified with 1 c.c. of 30 per cent. acetic acid, and the coloration obtained is compared with those given by known amounts of aluminium under the same conditions. If the quantity of aluminium present is more than 0·15 part per million, the colorations are best compared by observation through the sides of the cylinders.

W. P. S.

Organic Analysis.

Estimation of Ketonic Compounds and of β-Hydroxybutyric Acid in Diabetic Urine. H. Bierry and L. Moquet. (*Comptes rend.*, **1924**, **178**, 816–819.)—The processes of van Slyke and Hubbard may be applied to the estimation in urine of: (1) acetone present as such, together with that formed from acetylacetic acid during distillation, and (2) β-hydroxybutyric acid, which yields acetone when oxidised by means of sulphuric acid and dichromate. The apparatus used consists of a round-bottomed flask connected by sealed joints with a superposed tapped funnel and a vertical tube widened out like a pipette and bending over to connect with a condenser; this makes a tight joint at the bottom with a thistle funnel, by which the condensate is collected.

To defecate the urine, 25 c.c. are mixed with 100 c.c. of water in a 250 c.c. measuring flask and treated with 50 c.c. of 20 per cent. copper sulphate solution and then, gradually, with 10 per cent. milk of lime until the reaction is distinctly alkaline, this being indicated by the appearance of a blue colour; when filtered, the liquid should not reduce Fehling's solution. Twenty-five c.c. of the filtrate and a little pumice are placed in the flask, into which first 130 c.c. of distilled water and then 20 c.c. of dilute sulphuric acid (1:4 by vol.) are introduced through the funnel. Distillation is continued for 10 to 12 minutes, and 45 to 50 c.c. of distillate (*A*) are collected.

To the residual boiling liquid a mixture of 22 c.c. of sulphuric acid (1:1 by vol.) and 26 c.c. of a 0·15 per cent. solution of potassium dichromate (twice crystallised and fused before use) is added by way of the funnel. At intervals of 10 minutes during the distillation, three further quantities of 50 c.c. of the dichromate mixture are added, the distillation then continued for 15 minutes after the last addition, and 200 to 220 c.c. of distillate (*B*) collected.

The two distillates, which are collected with the precautions necessary to prevent loss of acetone, are mixed with the wash waters and redistilled, separately, in presence of 0·5 grm. of sodium peroxide, and the acetone is estimated iodimetrically in the resulting two distillates. In the case of *B*, the result is increased by 16 per cent. to compensate for loss of acetone under the conditions employed. One c.c. of 0·1 *N* iodine solution corresponds with 0·968 mgrm. of acetone or with 2·069 mgrms. of β-hydroxybutyric acid. T. H. P.

Sulpho-chromic Oxidation of Organic Substances and Coals in presence of Catalysts. Rôle of Mercury. D. Florentin. (*Bull. Soc. Chim.*, **1924**, **35**, 228–230.)—When heated with chromic and sulphuric acids in presence of a small quantity of mercury, all organic compounds, including coals, are completely oxidised, the sole gaseous product being mostly carbon dioxide; coke yields, under such conditions, also carbon monoxide, but in extremely small proportion. (*Cf.* ANALYST, **1922**, **405**, 530; **1923**, **37**, 398.) T. H. P.

Oxidation of Coal by Silver Dichromate and Sulphuric Acid. L. J. Simon. (*Comptes rend.*, 1924, **178**, 775–777.)—The results of investigations on the oxidation of coal indicate that coal consists of two components in intimate association. The one, which begins to gasify at a relatively low temperature, behaves towards chromic oxidising agents in the same way as aromatic hydrocarbons, gasification proceeding to completion when silver dichromate and sulphuric acid are used, but being only partial when chromic acid is used. The other constituent shows the behaviour of coke, gasification occurring only at about 100° C. and being incomplete under all the conditions investigated.

T. H. P.

Estimation of Glycerol in Crude Glycerins. Fachini and Somazzi. (*Bull. Ind. Olie e Grassi*, October, 1923; *Chem. Trade J.*, 1923, **73**, 702–703.)—The method originally applied to the estimation of glycerin in distilled glycerins (*Chem. Trade J.*, 1923, **73**, 127), which depended on the measurement of the amount of carbon dioxide produced by oxidation of dichromate, is now worked out for crude glycerins, which must be subjected to a preliminary purification. The most satisfactory preliminary treatment was found to be with basic lead acetate solution prepared by boiling a 10 per cent. solution of the salt with excess of litharge for 1 hour, and filtering under conditions eliminating contamination with carbon dioxide. The lead acetate solution eliminates proteins by coagulation, organic acids as insoluble salts, and alkaline carbonates as insoluble carbonates; and, on cooling to 0° C. for one hour, practically all chlorides as insoluble lead chloride. (Final traces of chlorides can be removed, if necessary, with a slight excess of silver carbonate solution.)

About 20 grms. of lye or 2 grms. of crude glycerin are weighed into a 250 c.c. flask, water added to a volume of 50 c.c., and then 5 c.c. of the basic lead acetate solution. After shaking and leaving for 30 minutes, the further addition of a small quantity of lead acetate solution should not produce any further precipitate, and, if a precipitate does form, the estimation must be begun again with a smaller quantity of sample. The contents of the flask are made up to 250 c.c. with water, and cooled in ice for at least one hour. After filtering, and when a temperature of 15° C. has again been reached, 25 c.c. are transferred to the oxidation flask, and the analysis continued as for pure glycerin, glycerol, and trimethylene-glycol being separately estimated. It has been found that glycerins of animal origin consist almost entirely of glycerol, whilst vegetable glycerins contain varying amounts of trimethylene glycol, and the method should prove useful in determining the origin of glycerins.

D. G. H.

Composition of Linseed Oil. A. Eibner and K. Schmidinger. (*Chem. Umschau.*, 1923, **30**, 293–302.)—A sample of linseed oil of Dutch origin was examined by modifications of the bromination method devised by Hehner and Mitchell (ANALYST, 1898, **23**, 310), and was found to contain the following constituents:—α-Linolenic acid, 21·1; an isomeric linolenic acid, 2·7; α-linolic acid, 17·0; oxy-acids, 0·5; saturated fatty acids, 8·2; glyceryl radical, 4·1; phytosterol, 1·0; unestimated (by difference), 46·2 per cent. Proof of the presence of an isomeric linolenic acid was obtained by brominating an ethereal solution of the oil, separating the insoluble bromide, removing the bromine from the filtrate, evaporating the ether by means of the pump, and treating the residue with petroleum spirit. The chilled solution yielded a deposit of the bromoglyceride of α-linolic acid, which was filtered off, and the filtrate was then debrominated, the esters saponified, and the fatty acids liberated dissolved in ether and again brominated. They now yielded 2·7 per cent. (calculated on the original oil) of a linolenic hexabromide (m.pt., 180° C.) bromine 63·03 per cent.

About 25 per cent. of the bromide of a mixed glyceride was obtained in crystalline form by dissolving the crude bromide (m.pt. 139° to 140° C.) in hot tetralin (tetrahydro-naphthalene, $C_{10}H_{12}$), adding boiling acetone until there were signs of precipitation, and then cooling the solution. The crystalline bromoglyceride melted at 155° to 156°, and contained: Br, 59·39; C, 32·1; and H, 4·6 per cent. This corresponded with the formula, $C_{57}H_{94}O_6Br_{16}$, and it was concluded that the compound was α-dilinolenic-α-linolic bromoglyceride. The other bromoglycerides present were not identified, but about 0·6 per cent. of a mixed glyceride was separated by treating the original linseed oil with nitrous acid; it was identified as di-elaido-palmitin. (*Cf.* Toms, ANALYST, 1924, 77.)

Oxidation of Lumbang and Linseed Oils and of the Principal Compounds in Lumbang Oil. A. P. West and A. I. de Leon. (*Philippine J. Sc.*, 1924, **24**, 123–142.)—Lumbang oil (obtained from *Aleurites moluccana*) had the following composition:—*Unsaturated glycerides*: linolenic, 6·5; linolic, 33·5; oleic, 57·0; *saturated glycerides*, 2·8 per cent. The oil was progressively oxidised by passing measured quantities of dried air through a small wash bottle containing the oil at a temperature of 75° C. and passing the excess of air through various absorption tubes. The operation was stopped at intervals for weighings to be made, and for comparative purposes linseed oil was similarly treated. At first (up to 30 hours) the percentage of apparent oxygen absorption, volatile products evolved and total oxygen absorption were found to be greater for lumbang oil than for linseed oil, but as oxidation continued the position was equalised (30 to 40 hours) and finally reversed. It is suggested that, on oxidation, autocatalysts are formed which tend to accelerate the velocity of oxygen absorption, whilst the saturated glycerides retard it. Since lumbang oil contains a smaller proportion of saturated glycerides than does linseed oil, the velocity of absorption is at first greater. Possibly in the case of linseed oil, after 30 hours a sufficient quantity of autocatalysts accumulates to overcome the retarding influence of the saturated glycerides and to exert a marked autocatalytic influence on the absorption velocity. The oxidised and unoxidised acids formed after blowing for definite periods at 75° C. were separated by Fahrion's method and found to be in the following proportions, oxidised acids being given first in each case:—Twenty hours (400 litres of air used), 7·13, 88·52; 40 hours (781 litres of air), 17·46, 79·07; 60 hours (1276 litres of air), 26·86, 68·66; 80 hours (1652 litres of air), 32·24, 62·12 per cent.

Constants of samples of lumbang and linseed oils blown for various periods of time at 75° C. were determined, and very similar results obtained. The experimental data obtained agree with the supposition that the absorption of oxygen takes place at the double bonds. Linolenic glyceride was oxidised more readily than linolic, and linolic more readily than oleic glyceride. D. G. H.

Chemical Factors Determining the Quality of Tobacco. V. Graham and R. H. Carr. (*J. Amer. Chem. Soc.*, 1924, **46**, 695–702.)—Although the change in the composition of tobacco produced by fertilisers is considerable, it is much less than that produced by different soils and different climates. For the comparative examination of tobacco the extract yielded to solvents affords most information. Six grms. of the samples were extracted with 60 c.c. of petroleum spirit, then with ether and then with alcohol, the nicotine being estimated in each extract by the official (Kissling) method. Cigar tobaccos have a small percentage extract and nicotine content, whilst smoking tobaccos have a high solubility in these three solvents. When the calcium content of the ash is high much of the nicotine is combined in a stable form, and so is insoluble in ether and other solvents; when a sample of powdered tobacco was mixed with calcium salts it was found that its nicotine became so fixed as to be extracted with difficulty, even by the official method. Acid phosphate fertilisers improve the aroma of the plants; much of the irritating effect of certain kinds of tobacco is due to loosely combined nicotine and volatile oils, both of which are easily soluble in petroleum spirit.

H. E. C.

Inorganic Analysis.

Potassium Permanganate as an Iodimetric Standard. J. M. Hendel. (*Zeitsch. anal. Chem.*, 1923, **63**, 321–324.)—The standardisation of thiosulphate solutions against permanganate gave results agreeing within 0·1 per cent. with those obtained by means of dichromate or iodine. About 35 c.c. of standard permanganate solution are run into a glass-stoppered flask containing 50 c.c. of 0·7 *N* hydrochloric or sulphuric acid, and 3 grms. of potassium iodide in 10 c.c. of water. (The iodide solution has been treated with aluminium amalgam to reduce any iodate). The stoppered flask is left in the dark for 2 to 5 minutes; the solution is diluted to 450 c.c. and titrated with thiosulphate in presence of starch. Atmospheric oxidation of the iodide during the titration is counteracted by the great dilution. In standardising against dichromate, 10 c.c. of the iodide solution are added to 200 c.c. of a solution containing about 0·13 grm. of potassium dichromate, and hydrochloric acid at 0·3 *N* concentration; the stoppered flask is left 10 minutes in the dark, and the solution, diluted to 800 c.c., is titrated as before.

W. R. S.

Estimation of Copper as Iodide. L. W. Winkler. (*Zeitsch. anal. Chem.*, 1923, **63**, 324–330.)—The neutral or weakly acid solution (100 c.c.) containing 0·1 to 0·01 grm. of copper is treated with one grm. of ammonium chloride and heated to boiling. The beaker is removed from the heat, and a 10 per cent.

solution of one grm. of potassium or sodium iodide added, followed by 10 per cent. sodium sulphite solution, drop by drop, until the precipitate is pure white; an excess of 5 drops is finally added. The liquid is again boiled for 1 to 2 minutes, and kept overnight in a dark place. The precipitate of cuprous iodide is filtered off, washed with 50 c.c. of cold water, dried for 2 hours at 130° C., and weighed. The following amounts are added to the weight, to allow for the solubility of the precipitate: 0·0013 for 0·4, 0·0011 for 0·2, 0·0010 for 0·1, and 0·0008 grm. for 0·01 grm. of cuprous iodide found. The method separates copper from magnesium, zinc, manganese, cobalt, and nickel. Lead interferes and must be removed as sulphate, and cadmium if present in very large amounts gives slightly low results. The process is recommended for the examination of blue vitriol, the commonest impurity in which is ferrous sulphate. An aliquot part of the solution equivalent to 0·5 grm. of the salt is treated as described above; the only modification consists in adding the slight excess of sodium sulphite and hydrochloric acid first, to be followed by the precipitant. W. R. S.

Schönbein's Reaction applied to the Micro-detection of Copper. H. and R. Imbert and P. Pilgrain. (*Bull. Soc. Chim.*, 1924, **35**, 60–64.)—When 3 or 4 drops of freshly prepared tincture of guaiacum resin and then, through a drawn-out tube, a few drops of 0·15 per cent. potassium cyanide solution are added to 5 to 10 c.c. of a neutral cupric solution, a distinct blue coloration is obtained even when the concentration of the copper is only 1 part in 2,000,000 of solution. Small quantities of copper may be separated from solution also by electrolysis, by means of platinum electrodes, in presence of nitric and sulphuric acids; with 10 c.c. of solution containing 0·001 grm. of copper per litre the copper deposited on the cathode after 45 to 50 minutes gives a slight reduction and blue coloration with molybdic acid reagent. These methods have been applied, with good results, to the detection and estimation of copper in distilled water, beans coloured with copper salts, and blood. T. H. P.

Estimation of Cadmium in Spelter and Zinc Ores. C. E. Barrs. (*J. Soc. Chem. Ind.*, 1924, **43**, 77–78 T.)—Ten grms. of spelter are dissolved in 30 c.c. of nitric acid and 100 c.c. of water, diluted to 250 c.c. and ammonia added until the precipitate of zinc hydroxide re-dissolves. The insoluble matter is filtered off and washed with 5 per cent. solution of ammonia; if it is bulky it is dissolved and re-precipitated. The filtrate is heated, an excess of 50 per cent. solution of sodium sulphide added, and the mixture, after being kept hot for an hour, is filtered; the unwashed precipitate is returned to the beaker and dissolved and the filter paper washed with nitric acid; then sulphuric acid is added and the solution evaporated until while fumes appear, after which it is cooled, diluted, and filtered. The insoluble matter is washed with dilute sulphuric acid, sufficient of which is added to make 20 per cent. in the filtrate, which is then warmed while hydrogen sulphide is passed through it to precipitate most of the copper. The precipitate is removed, washed with dilute sulphuric acid, and the filtrate diluted to at least 250 c.c. and again saturated with hydrogen sulphide to precipitate the cadmium. The cadmium sulphide is dissolved in a silica crucible with nitric acid, sulphated and weighed; then it is dissolved in dilute nitric acid, and any copper present is titrated by the iodide method, calculated as $CuSO_4$ and subtracted from the weight of the cadmium sulphate. Ores are dissolved in nitro-hydrochloric acid, the solution evaporated to dryness, taken up in hydrochloric acid, and the silica removed, then neutralised with ammonia, acidified with 1 c.c. of hydrochloric acid for each 100 c.c. of liquid, and a current of hydrogen sulphide is passed in. The precipitate is washed with dilute hydrochloric acid saturated with hydrogen sulphide, dissolved in nitric acid, ammonia and sodium sulphide are added and the analysis completed as above. H. E. C.

Separation and Estimation of Bismuth by Hydrolysis. G. Luff. (*Zeitsch. anal. Chem.*, 1923, **63**, 330–348.)—The nitrate solution (about 80 c.c.), free from ammonium nitrate, is neutralised with solid sodium bicarbonate until methyl orange just turns yellow. The liquid is acidified with a drop, and treated with 20 c.c., of *N* nitric acid. If the precipitate formed in the neutralisation does not re-dissolve completely, a little more acid is added. The clear liquid is diluted to 100 c.c. and boiled under reflux for one hour; 200 c.c. of water containing 0·5 c.c. less *N* sodium carbonate solution than the volume of *N* acid previously added is run in, drop by drop, through the condenser during the boiling. The cold liquid is filtered through a Gooch crucible; the precipitate of basic nitrate is washed with cold water, dried, and gently ignited to oxide. The process affords a good separation from lead, copper, and cadmium. W. R. S.

Separation of Zinc from Iron and Aluminium. E. G. R. Ardagh and G. R. Bongard. (*Ind. Eng. Chem.*, 1924, **16**, 297–299.)—Zinc may be separated from iron by means of ammonia and ammonium chloride in one precipitation when the following procedure is adopted. The solution, which may contain as much as 0·2 grm. of zinc and 0·4 grm. of iron, is treated with a few drops of hydrochloric acid and evaporated to a volume of about 5 c.c.; while this liquid residue is still warm 5 grms. of ammonium chloride are stirred into it and 10 c.c. of concentrated ammonia are added. The mixture is then diluted with 25 c.c. of water, the precipitate collected on a filter and washed with about 100 c.c. of ammoniacal ammonium chloride solution (5 grms. of ammonium chloride and 5 c.c. of concentrated ammonia per 100 c.c.) The filtrate is acidified with hydrochloric acid, diluted to 250 c.c., and the zinc titrated with standardised potassium ferrocyanide solution (22 grms. of ferrocyanide and 10 grms. of sodium thiosulphate per litre); uranium nitrate solution is used as indicator. The same method may be employed for the separation of zinc from aluminium, but the aluminium hydroxide requires rather more washing than does the ferric hydroxide. W. P. S.

Analysis of Zinc Ores. Use of Powdered Magnesium for the Removal of Copper and Lead. E. G. R. Ardagh and G. R. Bongard. (*Ind. Eng. Chem.*, 1924, **16**, 300–301.)—The use of powdered magnesium is recommended for the precipitation of copper and lead in hydrochloric acid solution; to ensure complete separation of zinc which may be present, the precipitated metals should be boiled with hydrochloric acid and re-precipitated with magnesium. The zinc in the filtrate from the copper-lead precipitate may be titrated directly with potassium ferrocyanide solution, since even large quantities of magnesium chloride do not interfere with the titration. If aluminium is used for the precipitation of the copper and lead it is necessary to remove the aluminium salts before the zinc is titrated. If the zinc solution contains not less than 7 c.c. of concentrated hydrochloric acid per 250 c.c. the presence of lead does not affect the titration of the zinc with ferrocyanide solution. W. P. S.

New Volumetric Method for Estimating Ammonium Salts. M. V. Auger. *Comptes rend.*, 1924, 178, 1081.)—The brown colour in the Nessler test does not appear till sufficient caustic alkali has been added to saturate the whole of the anion combined with the ammonium radicle. The author has applied this principle to the volumetric estimation of ammonium salts. The reagent is prepared by pouring on to 5 grms. of potassium iodide a saturated solution of mercuric chloride till the permanent precipitate forms. The whole is diluted to 100 c.c. and concentrated potassium iodide solution carefully added, drop by drop, till the precipitate disappears. A table is given showing the best quantity of this reagent to add to the solution of the ammonium compound; after the addition the mixture is titrated with sodium hydroxide solution till a permanent brown colour is produced. The titration has a sharp end-point, and is equally applicable to nitrate, sulphate or chloride. R. F. I.

Simultaneous Estimation of Two Halogens. H. Jahn. (*Chem. Zeit.*, 1924, 48, 150.)—The neutral solution of, *e.g.* chloride and bromide should occupy as small a bulk as possible; free acid is neutralised carefully with dilute ammonia. After addition of a few drops of chromate solution the liquid is titrated with 0·1 *N* silver solution, and the volume added is noted. An excess of silver solution and 5 c.c. of dilute nitric acid are then added; the solution is boiled, left to settle in the dark, and filtered through a Gooch crucible. The precipitate is washed with 3 per cent. nitric acid, dried at 120° C. in the dark, and weighed. The quantity of silver bromide in the mixed precipitate is given by the expression $4{\cdot}2234\,p - 5{\cdot}6116\,q$, in which p = weight of precipitate, and q = weight of silver used in the titration. W. R. S.

Reaction of Sulphur with Alkali and Alkaline-earth Hydroxides. H. V. Tartar and C. Z. Draves. (*J. Amer. Chem. Soc.*, 1924, 46, 574–581.)—When sulphur dissolves in aqueous solutions of sodium, potassium or barium hydroxide at the boiling point, polysulphides are first formed; on prolonged boiling these are partly reduced to di-sulphides and possibly mono-sulphides. The complexity of the polysulphide depends on the concentration of the reactants, and on the time and temperature. For the estimation of the mono-sulphide equivalent of the polysulphide in the presence of free alkali the A.O.A.C. method of iodine titration is unsatisfactory, but the following gives good results. The solution to be analysed is added, with constant stirring, to a mixture containing 50 c.c. of a buffer solution containing 8 grms. of borax and 5·5 grms. of boric acid per 100 c.c. and an excess of 0·1 *N* iodine solution over the mono-sulphide. Further 0·1 *N* iodine solution is added until all thiosulphhate has reacted and an iodine colour is obtained. From the total iodine is subtracted that equivalent to the thiosulphate; this is estimated in the filtrate, after precipitation of sulphides by by ammoniacal zinc solution, in the usual way. H. E. C.

Estimation of Water of Hydration, Water of Constitution, and Carbon Dioxide in Hydraulic Limes. J. A. Muller and E. Peytral. (*Bull. Soc. Chim.*, 1924, **35**, 220–225.)—The loss in weight of a hydraulic lime on calcination corresponds with the water of hydration and constitution, the carbon dioxide of the calcium carbonate, and any other carbon compounds present. It is determined by heating about 1 grm. of the material in a lightly covered platinum crucible gradually to bright redness in about 30 minutes, and then in a blowpipe flame at the highest temperature attainable for about 5 minutes; no further loss should occur when the residue is again heated for 5 minutes in the blowpipe flame.

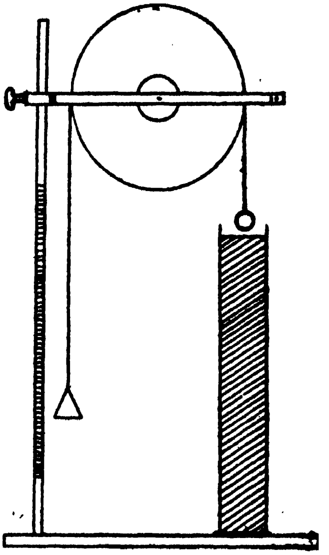
The water of constitution is composed of the water existing as acidic hydrogen or basic hydroxyl in the hydrosilicates and aluminates which, after the carbonates, the quartz sand, and the silicates unattackable by hydrochloric acid, form the principal components of hydraulic limes. The water of hydration is estimated by heating about 1 grm. of the lime in a platinum crucible at 185–190° C. to constant weight. The water of hydration and the water of constitution together are estimated by heating 4 to 5 grms. of the lime gradually to redness in a quartz glass test-tube about 12 mm. in diameter and 12 cm. in length. The tube, slightly inclined to the horizontal, is connected with a tared tube charged with pumice; its upper part is gently warmed to prevent condensation of the water vapour. After the lime has been heated for 15 minutes, the whole of the water of hydration and constitution is eliminated; the apparatus is then rapidly evacuated and disconnected from the pump, the pumice tube being detached and, when cold, weighed. The increase in weight represents the water of constitution and hydration. Subtraction of this increase from the loss on calcination gives the carbon dioxide, the result thus obtained agreeing closely with that obtained by decomposing the carbonates present in the lime with hydrochloric acid and measuring the carbon dioxide liberated. T. H. P.

Analysis of Gypsum and Gypsum Products. F. C. Welch. (*Ind. Eng. Chem.*, 1924, **16**, 238–241.)—The quantities of raw gypsum ($CaSO_4.2H_2O$), calcined gypum ($CaSO_4.\frac{1}{2}H_2O$) and anhydrite ($CaSO_4$) in a mixture containing any or all of these substances may be estimated as follows:—The sample is ground to pass a No. 48 sieve and the lime (CaO) and sulphuric acid (SO_3) are estimated gravimetrically. Hygroscopic moisture is then estimated by drying a portion of the sample in a current of air having a vapour pressure slightly greater than the dissociation pressure of gypsum at the temperature of the experiment. For instance, at 25° C., air of the required humidity may be obtained by passing it through sulphuric acid having a sp. gr. of 1·37 at 23° C. The drying is then completed by heating the sample in a U-tube at 200° C. while a current of dry air is passed through the tube and the water given off is absorbed in concentrated sulphuric acid and weighed. Let this quantity be termed W. The sample is cooled to ordinary temperature and a current of air passed over 25 N sulphuric acid is conducted through the U-tube until a constant weight is obtained. Let the quantity of water thus absorbed by the sample be termed w. If $W = w$, the water lost and gained was all present in calcined gypsum; when W is greater than w, the sample contained both gypsum and calcined gypsum, the amount of gypsum present being $172/27\ (W - w)$, and that of calcined gypsum $145/27\ (4w - W)$. If W is less than w, the sample contained both calcined gypsum and soluble anhydrite; in this case the amount of calcined gypsum is $145/9\ (W)$ and that of soluble anhydrite $136/9\ (w - W)$. W. P. S.

Physical Methods, Apparatus, etc.

Durability Tests of Glass. W. E. S. Turner. (*Pharm. J.*, 1924, **58**, 264–266.)—The action of boiling water on glass discs (average weight of each disc 80 grms.) of varying composition shows that loss in weight of the discs diminishes with the increase of lime present in the glass. Thus, starting with a sodium silicate base of molecular composition $6SiO_2 2Na_2O$, and progressively substituting calcium oxide for sodium oxide, a glass containing, for example, 0·21 per cent. of calcium oxide lost 24000·0 mgrms. per 100 square cm. on boiling in water for 6 hours, whilst with 6·26 per cent. of calcium oxide the loss was 2·5 mgrms., and with 11·68, 0·53 mgrms. Alkaline solutions have a very much more corrosive action on glass than acid. A satisfactory method for testing the suitability of reagent bottles is as follows:—The bottle is first thoroughly cleaned with weak acetic acid and distilled water, and dried. It is then suspended in a boiling water bath and a solution of narcotine hydrochloride (1 part of the salt dissolved in 1000 parts of distilled water in a platinum or good quality resistant glass vessel) added, the mouth plugged with cotton wool, and heating continued. Any alkali dissolved from the glass by the boiling water readily acts on the narcotine hydrochloride, causing precipitation of narcotine in the form of fine needles. With really good bottles an hour may elapse before precipitation begins, and the bottles may usually be regarded as satisfactory if no precipitate forms in 15 minutes. In the case of bottle glasses satisfactory durability is shown when not more than 18 per cent. of sodium oxide is present and less than about 8 per cent. of calcium oxide, but, generally speaking, it is simpler to insist that the glass comply with the durability test rather than to demand a specific composition, for some compositions outside these limits have been found satisfactory. Results of tests on eleven glasses of given composition are detailed. D. G. H.

A Simple Viscometer for Solutions of Resins, etc. K. Albert. (*Chem. Zeit.*, 1924, **38**, 181.)—Defects are pointed out in existing viscometers, especially in cases where the viscosity of unusually thick and dark liquids is required. The instrument described is on the principle of the falling sphere apparatus. A brass ball of 28·4 mm. diameter and weighing 103·06 grms. hangs over a wheel by means of a very thin thread of gut, at the other end of which is a balance-pan weighing 30·00 grms. The wheel, which has a diameter of 255 mm., is held in a fork which is clamped to a retort stand on which is fixed a cm. scale with two marks. A cylinder containing the liquid to be tested and of a diameter at least three times that of the ball is placed under the ball. To make the determination, suitable weights are placed on the balance-pan depending on the viscosity of the liquid. The two marks on the scale are arranged in such a way that the balance-pan passes the first mark only when the ball has fallen some cm. in the liquid. The time required for the pan to pass between the two marks is measured by means of a stop-watch, and the result expressed in cm. per second. The variation in several readings on castor oil and mixtures of this and resin-oil was not over 7 per cent. A castor oil with an absolute viscosity of 8·16 in Engler's viscometer at 20° C. gave a reading of 7·5 cm./sec. in this test. With this apparatus determinations are quickly made, and the parts can be readily cleaned, since the brass ball and gut only are affected.



R. F. I.

Spectrophotometric Determination of P_H Value and the Apparent Dissociation Constants of Indicators. W. C. Holmes. (*J. Amer. Chem. Soc.*, 1924, **46**, 627–631.)—The estimation of the P_H value of solutions is readily effected by the spectrophotometer by an empirical calibration of two ratios R_1 and R_2 with suitable indicators against known hydrogen electrode values. From the dissociation curves so obtained it is possible to calculate the dissociation constants. With an indicator having only one coloured form the ratio R_1 (which is the percentage colour transformation) is determined by measuring the relative absorption at the desired point and that under conditions of the maximum colour intensity. With two-colour indicators the ratio R_2 is measured; this is the intensity of absorption at two wave-lengths situated near the maxima of the two bands in question. Thus, for example, phenolphthalein has absorption bands at $460\mu\mu$ and $560\mu\mu$; the intensity at these wave lengths is measured and their ratio is plotted against known P_H values as determined by the hydrogen electrode. The P_H value of any solution can then be quickly found by measuring the ratio of the absorption at these wave lengths and referring to the curve. H. E. C.

Reviews.

A COMPREHENSIVE TREATISE ON INORGANIC AND THEORETICAL CHEMISTRY. Vol. IV. By J. W. MELLOR, D.Sc. Pp. 1074. LONDON: Longmans & Co. 1923. Price 3 guineas.

In less than two years we are in possession of the fourth volume of this invaluable treatise. The rapidity with which one volume follows another is astounding, and is all the more so when the excellence of the volumes is considered.

The present volume completes the study of the elements of the second group of the periodic classification. The first three chapters—203 pages—are devoted to the structure of matter, radium and radioactivity, and the architecture of the atom. The remaining chapters, extending over 846 pages, deal with the metals of Group IIB. With the exception of zinc and cadmium, the metals receive separate consideration.

The treatment accorded to the theories of the structure of the atom is exceedingly refreshing, especially in these days when so many speculations are rife. It is concise and the various hypotheses have been scrutinised in a rigidly logical manner. One is impressed by the enthusiasm with which Dr. Mellor discusses this subject, and this, no doubt, accounts for the large number of quotations which he has employed. We are reminded that the electronic theories of matter, unlike the older theories, are based on scientific evidence, although, as yet, that evidence scarcely can be regarded as anything other than circumstantial. Before proceeding to the general study of the theories, those subjects which give rise to this circumstantial evidence, namely, the physical properties of matter and radioactivity, are discussed. This section of the volume is concluded with a very apt paragraph, entitled "The Honesty of Science." We are told that although these "speculations probably make the best guess yet made about the ultimate constitution of matter," we are still "confronted with phantasmæ which would be banished at once if we were convinced that they were sterile conjectures and not pregnant hypotheses." In the reviewer's opinion we have in this volume the best general discussion of atomic theories so far written.

The remaining chapters of the volume deal with metals. Mention is made of the controversy which has gone on for a century over the name which should be assigned to the element beryllium. The author regards it as being "largely a question of temperament." This may, perhaps, explain why in papers which the reviewer submitted to two different societies the name was changed by their editors from beryllium to glucinum. Vauquelin, who discovered beryllia, did not assign any particular name, but referred to it as "*la terre du béril.*" He stated that its salts were sweet, and it was on this statement that the editor based the name "*la glucine.*" In reality, the salts are more tart than sweet, due to the hydrolysed acid, and this property can hardly be held as being peculiar to beryllium compounds. In the text of this volume the term beryllium is used, except in headings, when both names are given.

Dr. Mellor has the tendency to include, in the text, matter which seems to be contrary to general chemical principles. The mention of such substances as zinc cupric oxychloride, the formula of which is given as $(ZnCl_2.ZnO.2CuO)(ZnCl_2.3CuO).6H_2O$, and zinc dodecamminooxychloride, $2ZnCl_2.ZnO.12NH_3.4H_2O$, might well have been omitted (p. 546). The references should, of course, be inserted in the appropriate lists, and they merit only the briefest note, if any. It must be stated, however, that the author points out that there is little to show that these formulæ represent distinct chemical compounds, and that they require systematic study in the light of the phase rule before their individuality as compounds can be established. The hypothetical A, B and C forms of both beryllium and zinc hydroxides are dealt with without comment, although it is extremely dubious whether they really do exist. It is incorrect to say that Bleyer and Kaufmann, as is stated on page 225, obtained analytical data corresponding with the so-called acid hydroxide $H_2Be_2O_3$. Again, some criticism is surely wanting on the remarkable claim of Levi-Malvano that beryllium sulphate hexahydrate always crystallises from those solutions which were made from it, instead of the ordinary stable tetrahydrate (p. 237). Is it possible that at any given temperature the solution of one hydrate is fundamentally different from that prepared from another hydrate, such that the hydrate which separates is determined by the one which was used originally in making the solution, even though that hydrate may not be the stable solid phrase at that temperature ?

A few omissions appear to have been made from the extensive lists given. The reviewer failed to find references to six papers relating to metals discussed.

Apart from these minor points, the volume is well up to the standard of the previous volumes, and will be an indispensable asset to chemists and research students. HUBERT T S. BRITTON.

CONFÉRENCES DE CHIMIE MINÉRALE. MÉTAUX. By M. GUICHARD, Professeur à la Sorbonne. Third Edition. Pp. xxix.+355. Paris: Gauthier-Villars et Cie. 1924.

This text-book contains the author's course of lectures on metals delivered in the University of Paris to first-year students. It is not intended as a work of reference, as it contains a rather bald outline of the chemistry of the metals, only the most important compounds (oxides, hydroxides, and salts) being briefly described. On the other hand, much importance is attached to the application of the phase rule to salt solutions and metallic alloys, and numerous equilibrium diagrams are reproduced. The author has not much use for structural formulæ, and is in favour of using various modes of notation, such as CrO^3H^2O, CrO^4H^2, and $CrO^2(OH)^2$ for chromic acid; but the book seems to indicate a distinct partiality towards the old dualistic notation (*e.g.* AzO^3HAzH^3 for ammonium nitrate). A good many confusing formulæ appear in which two systems of notation are used without apparent reason, such as

$$SO^4H^2.2AzH^3(SO^4)^3Al^2.24H^2O \text{ (p. 70), and } CO^3UO^2CO^3Na^2 \text{ (p. 227).}$$

In the nomenclature of ammonium salts, the author is still keeping alive the antiquated designation "chlorhydrate d'ammoniaque." The reviewer remembers using Wurtz's *Traité* nearly thirty years ago while at school, and having to insert the term "chlorure d'ammonium" wherever the text-book lapsed into the old nomenclature. Contrary to Werner's proposals, the metallic ammines are termed "amines." The French chemical symbols are gradually drifting away from the international ones. "Az" for nitrogen is an old-established symbol; we are beginning to get familiar with "Tu" for tungsten. The present book uses "St" for strontium; we may live to see "Sb" changed into "An."

The salient feature of the work is its rejection of the Periodic system. The author holds that Mendeleeff's classification cannot be used in the teaching of chemistry, as its principle is too uncertain, and that it is certainly not the best classification. This *ex cathedra* pronouncement will cause the more curious among his students to inquire which classification the *conférencier* regards as the best; but his final opinion on a question that has a decisive bearing on the problem of the Unity of Matter is rendered by the following translation: "In conclusion it may be said that the classification of the elements cannot play a first-class rôle in science, for no very great confusion can result (*on ne peut faire un bien grand désordre*) with the small number of elements known—about a hundred—even if imperfectly classified" (p. 330). The principle of isomorphism as a means of classification appeals to the author, provided such elements only are grouped together as exhibit numerous cases of isomorphism among their compounds, giving what he explicitly terms artificial families (p. 330).

The book gives a table of the elements classified according to the existence, for each element, "of one or two characteristic oxides, that is, such as are easily obtained" (p. 328); while the old-fashioned subdivision into metals and metalloids has been maintained in the sequence in which the elements are described and discussed. The "metalloid metals" comprise the heavier elements of Groups IV. and V., with the exception of lead.

The reviewer is left with the impression that the present volume is hardly a product of the age of isotopes, X-ray spectra, and atomic physics.

W. R. SCHOELLER.

LEAD: ITS OCCURRENCE IN NATURE, THE MODES OF ITS EXTRACTION, ITS PROPERTIES AND USES, WITH SOME ACCOUNT OF ITS PRINCIPAL COMPOUNDS. By J. A. SMYTHE, Ph.D., D.Sc. Pp. 343. London: Longmans & Co. Price 16s. net.

This book is one of the Monographs on Industrial Chemistry edited by Sir Edward Thorpe.

As expressed in the preface, it has been the aim of the author to give, within somewhat restricted limits, a broad outline of the chemistry of lead.

Economy of words is an outstanding feature of the book. The short, well-balanced periods, together with the absence of superfluous presentation of fact, have resulted in an amazing amount of information on lead and the various views held thereon. The whole is contained in a space of 313 pages of well-written matter.

References are drawn from a particularly wide field, and the reader who may already possess an intimate knowledge of any one side of the subject will discover evidence of comprehensive selection from authorities with whom he is himself familiar. In this lies a test of general quality.

The preliminary chapter is aptly devoted to the history of the metal and those compounds known to the ancients.

Next follows a short summary of the occurrence of the various lead-bearing minerals, and in Chapter III. the author opens out the metallurgy of lead with a clear presentation of the chemistry of roasting and smelting. Here the influence of the principal associated minerals at working temperature, the behaviour of furnace gases, the velocity of reactions, and the nature of these phenomena are considered in the light of their bearing on the conditions of the furnace charge, its yield, and the slag production. There is probably little new in the description of blast furnace extraction, but the reader will find a good general survey of modern practice. The softening and desilverising, as well as the refining of the metal, are described as practised, and are considered in conjunction with the equilibrium diagrams of the alloys.

Under the subject of condensation of lead fume there is an excellent description of the Cottrell treatment; finally, so far as the metal is concerned, the most recent views and knowledge concerning its uses, properties, and alloys (including corrosion and the plumbo-solvency of soft waters) are brought within easy compass.

The remainder of the book is devoted to the compounds of lead. As is fitting, prominence is given to the oxides, their preparation, also to the production of white lead, and the theory of the processes. The complexity of this latter subject is well recognised by the author, who has been at pains to present the facts which are known, as well as to indicate the weakness of views which are not based upon the ascertained truth. This chapter is full of interest and is provocative of thought.

Other compounds of lead are dealt with in concise fashion and the book concludes with a chapter on lead-poisoning.

Altogether, the Editor of the series is to be congratulated upon his choice of Author for this Monograph, and the book can be offered with confidence for the use of those who seek knowledge of lead and its compounds.

GEO. R. THOMPSON.
JOHN MYERS.

QUANTITATIVE CHEMICAL ANALYSIS. By F. CLOWES and J. B. COLEMAN. 12th Edition. Pp. xxiv.+576. London: J. & A. Churchill. 1924. Price 18s. net.

The pleasure with which the many chemists whose early training was based on this book will hear of the appearance of yet another edition, will be clouded by the thought that it will be the last to receive the final impress of both authors, for Professor Clowes died before its publication.

When the first edition was published, in 1891, it was immediately recognised that the work had certain distinctive characteristics which made it particularly suitable as an introduction to the general methods of quantitative analysis. The arrangement, the style, the clearness of the directions, and the way in which numerous difficulties were forestalled, soon made the book a favourite both with teachers and students, and it is not surprising that new editions should have followed one another in rapid succession, until now the twelfth has been reached.

It is interesting to compare the first edition with this latest one. The original 409 pages have increased to 576 of a larger size, and, while the general arrangement has been maintained, many new methods of separation and details of analytical processes have been added, and the book now forms a trustworthy work of reference for practising analysts as well as a textbook for students.

In future editions space might, with advantage, be found for a description of colorimetric and electrometric methods of determining hydrogen ion concentration, and for sections on the uses of the microscope and spectroscope in chemical analysis.

By way of criticism it may be pointed out that attention should be called to the fact that the lime method of estimating halogens in organic substances is liable to give too low results.

Again, while a very good outline is given of some of the principal methods used in the examination of oils and fats, the deductions drawn from the constants of typical examples are only partly warranted, and make this branch of analysis seem a much more simple matter than it really is.

The question of terminology also calls for some remark. It is reasonable to restrict the term "estimation" to chemical analyses and "determination" to physical measurements, or to use "determination" for both operations, but there is nothing to be said for the indiscriminate use of the two terms.

Again, the authors state in their preface that the terms "hydrate" and "hydroxide" are used indifferently. This seems a pity, for it is now usual to make a sharp distinction in the application of the two words. This point is of more importance here than in many other cases, for the book is used by hundreds of students in colleges all over the world, and these will afterwards spread the use of the terminology of their student days.

Still, these are minor points which in no way detract from the general excellence of the book. Speaking from many years' experience of its practical value, the reviewer has no hesitation in saying that it is the best introduction to quantitative analysis with which he is acquainted. EDITOR.

JUNE, 1924. Vol. XLIX., No. 579.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, May 7th, 1924. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Johan Ernst Nyrop, Cand. Polyt. (Copenhagen University), and Clifford Padgett Thorpe, B.Sc. (Manc.).

Certificates were read for the second time in favour of:—Messrs. George William Clough, D.Sc. (Birm.), James Porter Shenton, F.I.C., and William Stanley Wood.

The following were elected members of the Society:—Messrs. John Joseph Bryant, Edgar Wilfred Deag, Harold Wilton Hewis, B.Sc. (Lond.), A.I.C., Ernest Wilfrid Jackson, F.I.C., Thomas McGrath, and Alfred Scholes, F.I.C.

The following papers were read:—"Estimation of Sugar in Urine by means of Fehling's Solution with Methylene Blue as Internal Indicator," by J. H. Lane, B.Sc., F.I.C., and L. Eynon, B.Sc., F.I.C.; "Simple Forms of Hydrogen Electrode," by J. J. Considene; "Note on the Recognition of Hydrogenated Oils," by K. A. Williams, B.Sc., and E. R. Bolton, F.I.C.; "Estimation of Copper and Tin and Copper-Tin Alloys," by A. T. Etheridge, M.B.E., B.Sc., F.I.C.; and "Notes on the Analysis of Milk Powders and Condensed Milks," by D. W. Steuart, B.Sc.

Report on the World's Dairy Congress.

By JOHN GOLDING, D.S.O., F.I.C.

(*Made at the Meeting, April* 2, 1924.)

Gentlemen,

As your delegate at the World's Dairy Congress, I have the honour to present the following report on my visit to the United States and Canada:

The Congress was a great success and was a triumph of organisation. Very great hospitality was shown to the delegates, and special facilities were offered for showing everything which was of special interest.

Upwards of 40 nationalities were represented, and the attendance from the United States of America included the leading scientific workers on every aspect of the industry.

The first meetings were held at Washington, where President Coolidge and other members of the Government welcomed the delegates on October 2nd and 3rd. The Sessions of the Congress were continued at Philadelphia on October 4th, and at Syracuse, New York, from the 5th to the 10th. From October 11th to the 13th excursions were arranged to Geneva and Cornell University, after which I visited New York. From October 18th to the 27th, I visited laboratories and colleges in America and Canada, leaving Montreal on October 27th. Other excursions were arranged by the Congress, but these I was unable to attend.

At Syracuse five Sessions were held simultaneously, and it was necessary to miss a great many of the 237 papers which were communicated. Most of these will, however, be published *in extenso*, and I only propose to refer briefly to a few of those which I had the privilege of attending.

A very excellent series of papers were contributed on the unidentified Dietary Factors or Vitamins, in which many of the leading authorities on this subject in America took part.

To the four previously defined vitamins or accessory food factors, a fifth was added by H. M. Evans (175). This new dietary factor, which he designated "X," was shown to be indispensable for reproduction. The factor is present in green leaves, cereals and fresh meat. Its further distribution in natural foods is being studied.

There is a definite, though low, quota of the needed substance X in a high proportion of milk fat; by feeding rats with milk fat in the proportion of 24 per cent. of the diet fertility may be secured in those animals. Commercial casein also contains the substance.

Discussions indicated a general acceptance of the reality and importance of these accessory factors in a complete and natural diet. No indication of their nature was suggested, and the term vitamin was provisionally accepted as a convenient designation of an unknown but essential dietary factor.

The occurrence of all the known vitamins in milk was accepted, but it was shown by R. A. Dutcher (179) and J. Golding (32) that the potency of milk in this respect varied considerably, and was dependent on the nature of the food supply of the cow. Other causes of variation had been studied, and C. Kennedy (33) concluded that drying and condensing milk will not destroy the vitamins it contains, though exposure to heat and oxidation in some processes of dry milk production may tend to diminish their content of vitamin *C*.

The value of milk as a food for children and as a cure for deficiency diseases, was illustrated by E. V. McCollum (70), who found an Institution where orphan children had been fed on white bread and a soup made from peas, beans, tuber and root vegetables, and muscle meats. For 48 of these children the diet was modified by the inclusion of a suitable amount of dried milk. The results fully corroborated the deductions drawn from experiments on animals.

La Fayette B. Mendel reviewed the causes of the value of milk as a food. He stated that the possible physiological properties of milk sugar remain to be elucidated, and spoke of the value of "sour milk" in this connection.

C. E. Bloch (74), of Copenhagen, reported medical evidence to show that blindness in children may be caused as a result of deficient nutrition.

H. C. Sherman discussed the optimum amount of milk for children, and as the result of a thorough experimental study of children receiving varying proportions of milk, concluded that for children of 3 to 14 years of age, one American quart (approximately ⅘ths of an English quart) per day must be given to insure the optimum storage of calcium and phosphorus.

The problem of the mineral requirements of milch cows formed the subject of three papers.

E. B. Forbes (176) gave results of experiments showing that fresh forage is more favourable to calcium storage than is dry hay, and hay cured in cocks under caps or covers is more favourable than is hay cured by direct exposure to the sun. As a practical method of giving nutrients to cows, free access was allowed to a mixture of 1 part of salt, and 4 parts of bone flour.

E. B. Meigs (177) reported the results of experiments extending over three years; these indicate that rations in which timothy hay constitutes the chief roughage, are deficient for dairy cows, not only in calcium, but also in some other unknown material which is plentifully present in alfalfa hay.

Oscar Erf (178) stated that 45 per cent. of the mineral salts can be washed out of alfalfa hay by heavy rains.

Several readers of papers advocated the use of dried milks for feeding infants, more especially in the tropics where satisfactory fresh milk is difficult to obtain.

The use of milk in bread-making was strongly advocated by R. M. Allen (34). This worker pointed out the greatly enhanced value of bread made with the addition of milk. Where whole milk is used, vitamin *A* is supplied, and there is an additional practical benefit in "shortening" the product. The new and well balanced bread was recommended made from an extract of the bran and wheat germ with milk and milk solids and additional calcium salts.

In the second paper Allen points out the necessity for legislative control in the sale of milk-breads. The questions of the amount of milk to be required before the loaf may be called milk bread and what descriptive terminology shall be allowed when skim milk solids are used were discussed. A committee considering this subject suggested, as a standard, that at least one-third of the liquid ingredients in the manufacture of milk-bread shall be whole milk.

C. A. Glabau (35) spoke with great enthusiasm of the possibility of finding a market for skim milk solids in the baking industry. He said that the baker consumes 40,000,000 barrels of flour per year, which furnishes a market of about 11 pounds of skim milk solids per barrel of flour, or about 15½ pounds of whole milk solids per barrel of flour. The vast economical importance of utilising dairy by-products in the baking industry, and by so doing increasing the nutritive value of the "staff of life," was generally acknowledged as a highly desirable goal by those who took part in the discussion.

Two papers were read on the selection of metals for dairy equipment by O. F. Hunziker and R. Seligman. It was generally concluded that this subject requires more investigation, and the introduction of a variety of new metals in the manufacture of expensive dairy outfit, necessitates a more complete knowledge of the action of milk on these metals and the effects produced by the metals on milk. This subject was also brought out in papers dealing with dried milks.

Much information about chemical and analytical problems was gleaned by visits to laboratories and colleges. With an introduction from Mr. Henry C. Wallace, Secretary of the United States Dept. of Agriculture, I visited the Bureau of Chemistry, Washington, D.C. I had the pleasure of meeting the new Chief, Dr. C. A. Browne. The acting Chief, now Director of the Regulatory Department, Mr. W. G. Campbell; the Director of Research, Dr. Bell; the Assistant Chief of the Bureau, Dr. W. W. Skinner; and Mr. K. J. Keister, in charge of Milk Analysis.

Among the many activities of the Bureau, the investigation and development of processes for the utilisation of agricultural products and by-products were of special interest. Among these were the utilisation of dried milk, dried separated milk, and dried butter milk in bread-making. In this connection the Regulatory Department seemed to be in some difficulty in enforcing and controlling suitable trade designations for the products which are now important articles of food. The practical advantages of milk fat in "shortening" the bread and the better balanced food which resulted are certainly worthy of recognition. The less expensive by-products of the butter industry also add to the nutritive value of the bread, but when I discovered in Canada a soya bean product which was also being proposed for making "milk-bread," the need for suitable designation and analytical control became more apparent.

A list of some of the publications issued under the auspices of the Department is appended :

U.S. DEPT. AGRICULTURE LIBRARY. No. 6. Bibliographical Contributions. Partial List of Publications on Dairying issued in U.S.A. 1900 to June, 1923.

Contents:—Part I., Publications of the State Agricultural Experiment Stations, pp. 4–75; Part II., Publications of the U.S. Dept. of Agriculture, 1900 to June, 1923, pp. 76–97; Part III., Books issued or revised since 1908, pp. 99–103; Part IV., Periodicals, pp. 104 to 107; Subject List, pp. 108–236.

STANDARD METHODS OF MILK ANALYSIS of the American Public Health Association and the Association of Official Agricultural Chemists. Fourth Edition. 1923.

REPORT OF THE CHEMIST. U.S. DEPT. OF AGRICULTURE, for the year ending June 30, 1922.

A HANDBOOK OF DAIRY STATISTICS. By T. R. Pirtle. U.S. Dept. of Agriculture. June, 1922.

THE DAIRY PRODUCE ACT AND REGULATIONS. Acts, Orders and Regulations, No. 6. Dept. of Agriculture, Canada. 1923.

DISCUSSION.

Mr. E. R. BOLTON expressed great satisfaction that Capt. Golding had been able to represent the Society as a delegate at the world's Dairy Congress. He was interested in Miss Kennedy's view that the process of drying milk did not destroy vitamins. "Dried" Milk was an insufficient description: milk might be dried by three distinct types of processes, and he would like to know whether Miss Kennedy's statement referred to one type only or covered all types.

Dr. J. C. **DRUMMOND** said that this question of the temperature to which vitamins could be heated was constantly coming up, and was one that it was quite impossible to answer without knowing particulars of the process. No work had, so far, been published in which samples had been examined before and after drying by any particular process. The destruction of vitamins was brought about by oxidation, and the conditions that led to oxidation being rise of temperature, length of exposure to that temperature, and degree to which the product was aerated, it was evident that, by a combination of these factors, one got destruction. There were milk products on the market which had not been satisfactorily prepared, but if dried milk was in a fresh condition it appeared to have as good vitamin value as an average sample of fresh milk.

Mr. A. **CHASTON CHAPMAN** said that there appeared to be some evidence that the vitamins in milk were not destroyed in the ordinary processes of drying, and if there was any truth in the statement which had recently appeared in the press that one of the vitamins was a crystalline substance melting at 233° C., it would be reasonable to suppose that these substances were tolerably resistant to the destructive effect of heat.

Mr. G. **RUDD THOMPSON** said that evidently one more terror had been added to life in a fifth accessory food factor. He had carried out experiments, extending over three years, on hay, and from some slight investigations upon alfalfa, he would not be surprised to find the missing ingredient to be fluorine. He considered dried milk would be invaluable in the Tropics, where Europeans often found it almost impossible to get satisfactory milk. He hoped that the suggestion in Mr. Allen's paper of legislative control in the sale of milk-bread for the percentage of milk-solids would not be introduced into this country. As regards the selection of metals for use in dairies, he had, some time ago, conducted experiments on behalf of some dairy farmers, and had been surprised by the corrosive effect that really normal milks had upon certain aluminium-copper alloys. He thought there was a field for investigation in dairy utensils. He enquired if America was more up-to-date in the matter of dairying than we were in this country, and, if so, why it was.

Dr. J. A. **VOELCKER** said that he was interested in Capt. Golding's remarks on practical matters in connection with dairying in America, and enquired how the position stood with regard to scientific dairying, such, for instance, as research laboratories.

Mr. H. **JEPHCOTT** said that he had been present at the Congress when Dr. Kennedy's paper was read. The dried milk in which Dr. Kennedy stated vitamin destruction had not occurred had been prepared by the roller process, and not in a vacuum.

Capt. J. **GOLDING**, referring to Mr. Bolton's question, said that Dr. Kennedy spoke of milk dried or condensed by different processes. She concluded that very little destruction, even of vitamin C, took place when the water was rapidly removed by modern methods.

Rapidity of drying on a roller, low temperature in a spray process, and absence of air in a vacuum process, all tended to prevent the oxidisation which resulted in destruction of the vitamins. The fat-soluble factors were more thermostable than the vitamin C.

Dr. Drummond's remarks summarised the position and were, Captain Golding believed, in agreement with the views of American workers, who fully appreciated the variation in milks due to the feeding of the cows.

G. W. Cavanaugh, R. A. Dutcher and J. S. Hall (*J. Inf. Dis. of Children*, **25**, **498**, **1923**) had recently taken this fact of variation into account in their work on a spray-dried milk. They concluded that milk powdered by the spray process seemed to possess as great anti-scorbutic properties as the raw milk from which it was made.

Referring to the President's question as to whether American dairy methods were more up-to-date than British, the speaker said that there was no doubt that on seeing American methods, one learnt a great deal of what to follow and of what to avoid. For instance, it was curious to see a brewery bottle-washing machine adapted to dairy-bottle washing, and the difference of opinion evinced in the fundamental principles involved in the apparatus. On the other hand, the Americans understood the use of ice for the carriage of milk in a way which we at present did not. The Americans were fortunate in having access to greater financial resources than were available in this country, with the result that their educational Dairy buildings were large and well equipped.

Cream Cheese.

By T. R. HODGSON, M.A., F.I.C.

(*Read at the Meeting, March* 5, 1924.)

An attempt was recently made by the Holborn Borough Council to set up a standard for fat in cream cheese (Analyst, 1923, **48**, 383); it was apparently the intention of the Council to insist that cream cheese should contain at least 20 per cent. of fat; the prosecution, according to the report, expected cream cheese to be made from cream, and the defence contended that it could be made from skimmed milk.

This attempt to set up a minimum standard for fat would appear to be very ill-advised for two reasons:—(1) The decision carries no weight outside London, because, although at the police court, the decision was in favour of the prosecution, an appeal from the magistrates' decision, being a question of fact and not of law, is taken to the Quarter Sessions and the decision of the Chairman of the London Quarter Sessions has no effect outside the area of his own Court's jurisdiction; the provincial Analyst is therefore left in this position that, as the appeal was allowed, the case can be quoted against him in similar proceedings, whereas if the appeal had been dismissed it could not have been quoted in his favour. (2) The proposed standard leaves out of account altogether the composition of the original milk from which the cream cheese is made; that this is most important is seen from the United States Standard, under which cream cheese must be made from cream or from milk containing not less than 6 per cent. of fat; this standard obviously does away with the contentions of both the prosecution and the defence, as the minimum requirements are that it should be made from whole milk; as it is quite possible to make cream cheese which contains less than 20 per cent. of fat, from whole milk, and it is also possible to make cream cheese, containing more than 20 per cent. of fat, from partially skimmed milk.

In any similar prosecution, it would be much more reasonable to charge the vendor with selling cream cheese made from milk partially deprived of its fat;

this raises an altogether different question, namely, as to whether it is legitimate to manufacture cream cheese from skimmed or partially skimmed milk, and further, any appeal from a police court decision, being on a point of law, would necessarily have to be taken to the High Court of Justice, with the result that the final decision would be binding on the whole country and not on London alone.

The method of deciding on the results of an analysis, as to whether a sample is made from skimmed or partially skimmed milk, is very simple; Cribb has shown (ANALYST, 1909, **34**, 45) that in a genuine cream cheese the percentage of fat should exceed the percentage of protein and, further, Richmond (ANALYST, 1919, **44**, 202) has shown that the composition of the original milk can be calculated from the formulæ:

$$\text{Fat in original milk} \quad \frac{100\text{F}}{35{\cdot}4\text{P} \times \text{F}} + 0{\cdot}25.$$

$$\text{Solids-not-fat in original milk} \quad \frac{333\text{P}}{35{\cdot}4\text{P} \times \text{F}}.$$

For the purpose of testing the above formulæ, 26 samples of cream cheese were examined, of which 19 were made in the laboratory from milk of known composition, and 7 were purchased from retail shops. Analysis of these samples gave following results:—

	Fat Per Cent.	Proteins Per Cent.	Water Per Cent.	Lactose Per Cent.	Mineral Matter Per Cent.
1	66·03	9·15	23·80	0·33	0·69
2	46·37	12·79	37·63	2·00	1·21
3	45·17	13·84	37·39	0·79	2·81
4	37·22	18·95	42·20	0·91	0·72
5	28·23	27·72	38·73	1·21	4·11
6	28·02	28·90	38·18	0·79	4·11
7	23·71	15·94	55·00	1·84	3·51
8	22·53	16·45	58·09	2·05	0·88
9	21·34	10·42	66·15	0·76	1·33
10	19·13	18·62	57·50	1·32	3·43
11	17·38	17·15	62·61	1·93	0·93
12	16·42	15·31	65·42	1·54	1·31
13	13·60	21·76	61·08	2·07	1·49
14	13·21	18·52	65·17	1·99	1·11
15	12·23	10·92	72·53	1·11	3·21
16	6·43	15·85	74·59	1·41	1·72
17	5·00	23·91	67·13	0·63	3·33
18	1·97	22·13	72·45	1·77	1·68
19	1·06	24·29	71·12	0·72	2·81
20	76·15	3·26	18·85	0·53	1·21
21	26·42	24·71	44·80	1·12	2·95
22	22·53	19·03	54·32	1·33	2·79
23	20·9	28·3	46·4	1·6	2·8
24	15·7	26·4	52·7	1·6	3·6
25	11·8	17·7	66·5	1·5	2·5
26	8·2	24·9	63·2	1·5	2·2

Nos. 1 to 9 were made in the laboratory and Nos. 10 to 26 were purchased in the ordinary course of business.

Comparing the above analyses with the proposed standard, Nos. 1 to 9 and 20 to 23 comply with the standard and Nos. 10 to 19 and 24 to 26 fail to comply and would therefore be reported upon as adulterated; but the actual and calculated compositions of the original milks were as follows:

	ACTUAL.		CALCULATED.	
	Fat Per Cent.	Solids-not-fat Per Cent.	Fat Per Cent.	Solids-not-fat Per Cent.
1	17·19	7·80	17·18	7·81
2	9·63	8·64	9·54	8·53
3	8·75	8·55	8·69	8·63
4	5·56	9·00	5·51	8·91
5	3·09	9·16	3·04	9·14
6	2·89	8·86	2·92	8·88
7	4·33	9·13	4·28	9·03
8	4·00	9·20	3·98	9·06
9	5·77	8·85	5·72	8·89
10	3·09	9·16	3·07	9·14
11	3·12	9·16	3·03	9·30
12	3·22	9·14	3·19	9·13
13	2·00	9·31	1·98	9·24
14	2·00	9·31	2·22	9·22
15	3·41	9·45	3·39	9·33
16	1·36	9·39	1·38	9·30
17	0·85	9·45	0·83	9·34
18	0·48	9·41	0·50	9·38
19	0·40	9·41	0·37	9·39
20	—	—	40·21	5·69
21	—	—	3·26	9·37
22	—	—	3·49	9·10
23	—	—	2·27	9·21
24	—	—	2·10	9·23
25	—	—	2·10	9·23
26	—	—	1·18	9·40

In the above table the calculated fat and solids-not-fat were obtained by the use of Richmond's formulæ.

Now if we consider the above figures, we find that Nos. 1 to 5, 7 to 12 and 15 are made from whole milk and, yet, under the proposed standard, Nos. 10, 11, 12, and 15 would be condemned as adulterated; the unfairness of this standard may be judged from a consideration of Nos. 5 and 10; these two cheeses were made from the same milk and, yet, under the standard, No. 10 would be condemned but No. 5 would be passed as genuine; also No. 6 would be passed as genuine, although made from a milk slightly deficient in fat.

By applying Cribb's formula to the above results, we find that Nos. 1 to 5, 7 to 12, and 15 would be passed as genuine, and Nos. 6, 13, 14 and 16 to 19 classed as adulterated; this actually corresponds with the facts.

Of samples Nos. 20 to 26, the proposed standard would pass Nos. 20, 21, 22, and 23, and would condemn Nos. 24, 25 and 26; Cribb's formula would pass Nos. 20, 21 and 22 and condemn Nos. 23, 24, 25, and 26. It must, therefore, be obvious from the above that it is quite arbitrary and contrary to the obvious facts to attempt to set up any standard for fat which has no reference to the protein content.

What is Bondon Cheese?

By G. D. ELSDON, B.Sc., F.I.C.

(*Read at the Meeting, March 5, 1924.*)

During October last a dairyman in the Borough of Salford was fined £5 for selling as "cream cheese" a cheese which contained only 5 per cent. of fat. The cheese was supplied to the vendor under the description of "Bondon," the labels, however, being removed before sale. Although the court upheld the opinion that a cheese such as this should not be sold as "cream cheese," the question as to whether such a cheese may be lawfully described as "Bondon" remains unanswered. A case recently heard in the Appeal Court in regard to a somewhat similar matter affords us no help as, owing to a conflict of evidence, the matter was fought out on a side issue.

In attempting to arrive at a decision the writer has endeavoured to obtain evidence from various sources; a large amount of material has been collected, and an attempt is now made to summarise the information that has been obtained.

Cribb (ANALYST, 1909, 34, 45) makes the following comments:

> "It is noteworthy that the produce of the well-known English dairy companies all contained upwards of 69 per cent. of fat, while the worst cases are to be found amongst the varieties labelled as 'Bondon,' one at least of which was made in this country. The true 'Bondon' cheese as made in France, was, and possibly still is, made from whole milk; if it is to be sold here as cream cheese, the least that can be expected is that it should have the composition of a whole-milk cheese. 'If the Bondon cheese is to be accepted as a cream cheese' then the fat should be well over 20 per cent. . . .'' If, however, the Bondon is to be excluded from this category, then the standard suggested by Moor (at least 40 per cent. of fat) would be a perfectly reasonable one."

Richmond, *Dairy Chemistry*, 3rd edition, gives three analyses of Bondon cheeses by various observers, which vary from 20·8 to 41·9 per cent. of fat, whilst he also reproduces the figures due to Cribb above and describes them as "Bondon?" In a letter to the writer, however, he states that, "Neufchâtel cheese, which is frequently called Bondon, is a product chiefly made in the department of Seine Inférieure in France, the origin probably being in the town of Neufchâtel. It is a soft cheese and is usually made from whole milk, but there are, as a matter of fact, several varieties. First of all there is the Neufchâtel "Double crême dit Bondon" cheese made from whole milk to which a proportion of cream is added, then there is the whole milk cheese or Neufchâtel cheese "à tout bien," this is the normal Neufchâtel or Bondon cheese and is, I believe, a standard variety. There is, however, a cheese made from skimmed milk or Neufchâtel "maigre" or "à la pic," this is, or has been, also sold as Bondon, and I believe would be distinguished from the standard Bondon cheese, partly by the name, but especially by the price at which it is sold.

"A Bondon cheese containing 5 per cent. of fat would certainly come under the category of the skimmed milk cheese. I have no evidence as to what the sales of the skimmed milk cheese are in Normandy, as, though I have been to several places in the department of Seine Inférieure, I have never bought, nor have noticed, any Bondon skimmed cheese exposed for sale.

"My own impression is that the skimmed milk cheese is not a very largely sold article. So far as England is concerned, I doubt whether there is any understanding of what Bondon cheese should be, and I think that as long as the purchaser gets a soft cheese of the characteristic Neufchâtel type, he would not be prejudiced if the skimmed milk cheese were sold to him so long as it was sold at a low price."

The writer has discussed this matter with several prominent Public Analysts in the North of England, and they were unanimously of opinion that such a cheese ought certainly not to be sold under the name "Bondon" unless the purchaser is distinctly notified that it is a skimmed milk cheese.

The authorities of the municipal laboratory, Paris, on being approached on this matter, replied in the following manner:

> "The cheese called 'Bondon' which is used in France should normally contain 20 to 25 per cent. of fat, although standards for fat in cheese have only been fixed in the case of 'Camembert.' I have every reason to believe that a manufacturer who placed on the market a Bondon containing only 5 per cent. of fat would be liable to be prosecuted if not for fraud at least for deceiving the public as to the quality of the goods."

The writer has approached two prominent members of the Dairy Industry in the North Midland area. The first stated that, in his opinion, a Bondon cheese should be a whole milk cheese containing therefore 20–30 per cent. of fat (the percentage of fat in a soft cheese tends to be lower than that in a hard cheese on account of the increased proportion of water), this opinion being supported by the fact that the produce of his firm contained 28 per cent. of fat as determined in this laboratory. The second expressed the opinion that "If I were buying a Bondon cheese I should expect it to be a whole milk cheese and not a skimmed milk one."

Inquiries have been forwarded to the Directors of nine Agricultural Colleges in this country. The general idea which seems to be held at these colleges, as shown by the replies that have been received, is that the original Bondon cheese was made from whole milk sometimes enriched with cream, but that in more recent years the practice has grown up of sending out cheese under this description which has been made from all kinds of milks ranging down almost to separated milks. The opinion is also expressed that the cheese made from separated milk is distinctly inferior, and that the best kinds are still made from whole milk.

An important firm in the Dairy Industry, one of those firms who consider that the preparation of Bondon cheese is a legitimate manner of utilising skimmed milk, write in the following way:

> "We cannot, however, agree with you that Bondon cheese made from skim milk should not be sold as such, as for a considerable number of years (over 20 to our knowledge), cheese made in this manner from skim milk with even less than 5 per cent. fat, has been sold under the name 'Bondon,' and we think if you refer to any text book you will find confirmation of this. Bondon cheese is made from whole milk, skim milk and partially skim milk both in Normandy (the home of Bondon cheese) and in this country, evidence of this being unlimited, indeed this is the first time we have heard this fact either disputed or queried in any way."

Reviewing the whole of this evidence as impartially as possible, two main facts would seem to be established: (1) That Bondon cheese was originally a whole milk cheese, and that it is still so regarded by many. (2) That an important section of the dairy industry are now making cheese from skimmed milk which they describe as "Bondon," and which practice they consider to be legitimate. As in nearly every other case, the last factor which the trade seem to take into consideration is the opinion, knowledge and requirements of the public, and it seems to the writer that it is grossly unfair to that section of the trade which is selling a good class article to allow another section of the trade to produce an inferior article under the same descrpition. It is quite true that, in some cases, the inferior article is sold at a slightly lower price, but this fact is not usually considered to be a legitimate defence in any case of misdescription, and, in the interests of both consumer and trade, it would appear to be most desirable that some definite standard for Bondon cheese should be laid down or, alternatively, that where any soft cheese is not made from whole milk the fact should be plainly declared to the purchaser by a distinctive label

In the opinion of the writer "Bondon" cheese should be a whole milk cheese and should certainly not contain less than 20 per cent. of fat, whilst possibly 25 per cent. might be a better standard, although more definite information would have to be obtained before this point could be finally settled. The fact that skimmed milk cheese has been sold as "Bondon" for the last twenty or even fifty years should surely be no argument at all, if Bondon cheese is really a whole milk cheese, because some firms are still selling a whole milk cheese as Bondon and declare this to be necessary. It should not be possible to allow any firm or firms to plead "Trade custom" merely because they have been selling for years an article alleged to be adulterated without the matter being discovered or brought to light in the courts.

A word or two might be said here in regard to the defence which is so often brought forward by defendants in the case of proceedings under the Food and Drugs Acts that they have been selling similar goods for years, and that no complaint has previously been made—drawing the conclusion that matters have always been satisfactory up to the time in question. The only test that is of any value in such cases is, of course, the percentage of samples, taken under the Acts which have been found to be genuine and adulterated respectively.

DISCUSSION.

Dr. G. **MONIER-WILLIAMS** said that in suggesting a standard for cheese it appeared to be an unnecessary complication to take account of the water content. What was really required was to know whether a cheese had been made from cream, whole milk, partially skimmed milk, or separated milk. The ratio of fat to protein in the original milk was the controlling factor as regards the composition of the cheese, and this had been found in Holland to be remarkably constant. Swaving had given a series of figures in which the fat content of the original whole milk varied from 2·55 to 4·01 per cent., the resulting cheeses varying only from 47·9 to 53·3 per cent. of fat. In America the standard for a whole milk cheese was 50 per cent. of fat on the dry solids. In Holland this standard could, apparently, not be reached, and 45 per cent. was laid down as the figure for whole milk cheese. Anything under 20 per cent. of fat on the dry solids had to be labelled as skim milk cheese.

Mr. **HINKS** said that he agreed with the last speaker. Cream cheese should be made from cream, otherwise the meaning of the term had entirely gone. Apart from the actual fat content of the cheese, the fact that the "creaminess" might be produced by water should also be considered. He was inclined to think that some of the "cream cheeses" referred to by the author must be skim milk churned with water. In a cheese containing 80 per cent. of water the ratio of fat to protein would be very small. Was there a double standard for cream cheeses similar to the condensed milk standard? It was absurd to have a "cream cheese" containing only 5 per cent. of fat. He thought it advisable that a double standard should be set up.

Dr. **DYER** suggested that the term "cheese" was applied to something you obtained in the form of a slab; the term "cream cheese" was loosely applied to all soft cheeses and, as popularly used, had lost to a large extent, its proper meaning.

Mr. C. L. **CLAREMONT** said that the term "cream cheese," as used by the author, did not mean made from cream, but merely meant of a creamy consistence. It might be made by warming milk and adding rennet.

Mr. E. M. **HAWKINS** thought that surely the nutritive value of the cheese should be considered; a buyer did not expect to have to pay for "water standing up" at the price of a cream cheese. If that was the case, the manufacturers of these cheeses must be making a handsome profit. Were these cheeses made, possibly, from whole milk emulsified?

Dr. J. A. **VOELCKER** said that the authors' definitions of cream and soft cheeses were very different from those which ruled at Agricultural Shows. At the latter there were separate classes for "Soft Cheeses made from whole milk" and for "Cream Cheeses made from cream without the addition of rennet." It was dealing unfairly both with the farmer and the public to call a "soft" cheese a "cream cheese," and he thought that it was time that something was done to secure uniformity of action. From the agriculturist's point of view "cream cheese" was a cheese made from cream and nothing else.

Mr. A. **MORE** said that when doing experiments with rennet and milk a by-product was obtained, which, if put in a cloth and allowed to drain, was described as cream cheese. The term "cream cheese" resembled some others which were misleading; there was "evaporated cream" which was not derived from cream, "egg powder" which never saw eggs; also several kinds of soft cheese, much advertised, and generally considered to be cream cheese, were probably not made from cream at all.

Mr. T. **MCLACHLAN** said that the dictionary defined "Bondon" cheese as one made at Neufchatel in the shape of a "bondon" or bung. If the French understood the term "Bondon Cheese" to mean a cheese of a particular shape made in a particular place, surely it was quite irregular to sell "Bondon Cheese," not made in Neufchatel, as such in England.

The Estimation of the Inorganic Impurities in Gelatin.

BY S. R. TROTMAN, M.A., F.I.C., AND R. W. SUTTON, B.Sc., A.I.C.

THERE is no standard of purity for gelatin sold for food purposes in the United Kingdom, but in the United States of America limits are prescribed by the Food Laws which must not be exceeded. These are, in parts per million:

Arsenious oxide, 1·4; sulphur dioxide, 350; lead, 50; copper, 30; and zinc, 100. Aluminium, iron and calcium, also commonly present, are not subject to regulation.

The estimation of these impurities is not easy. The results obtained by different chemists do not agree so closely as might be expected. They appear to be affected by the method used for analysis. The authors have had a large experience in the analysis of gelatins for export, and have examined some of the published methods very carefully.

The following brief details are given:

1. ARSENIOUS OXIDE.—Destruction of organic matter appears to be essential. For this purpose, magnesium nitrate solution, as recommended by Wilkie, *J. Soc. Chem. Ind.*, 1923, **42**, 63, is quite satisfactory. The electrolytic apparatus of Monier-Williams (ANALYST, 1923, **48**, 112) and that of Wilkie (*loc. cit.*), are used regularly, but the authors prefer standard mirrors to the stains recommended by Wilkie.

2. SULPHUR DIOXIDE.—The only reliable method is to distil, by means of steam, into excess of bromine water, and weigh the sulphuric acid produced, as barium sulphate (*cf.* Chapman, ANALYST, 1922, **47**, 204). Distillation into iodine solution and titration is unreliable, since gelatins containing no sulphur dioxide give a distillate which reduces iodine. But a gelatin free from sulphur dioxide gives no sulphuric acid when distilled with steam, even if distillation is continued for two hours, while the whole of the sulphur dioxide comes over in about 20 minutes. It should be noted that discrepancies in results occur sometimes through want of uniformity in determining (*a*) free sulphur dioxide, (*b*) total sulphur dioxide after treatment with acid.

3. THE ESTIMATION OF METALS.—This is rendered difficult by the relative amount and nature of the organic matter present. Thirty grms. of gelatin is the smallest quantity which can be used for reliable results. Hertwig (*J. Soc. Chem. Ind.*, 1923, **42**, 1034 A) destroys organic matter by incineration. This is very tedious and liable to result in loss of both lead and zinc. The authors prefer the method of Jamieson (ANALYST, 1919, **44**, 124). From 30 to 50 grms. of the gelatin are soaked overnight in cold water. About 25 c.c. of strong hydrochloric acid are added, and the mixture is heated on the water bath for two hours to hydrolyse

the gelatin. The liquid is then cooled, made alkaline with ammonia, and subjected to the action of a stream of hydrogen sulphide, or treated with sodium sulphide solution (*cf.* Lander, ANALYST, 1908, 33, 450). The precipitated sulphides are coagulated by heating in boiling water, collected on a Gooch filter, washed with hydrogen sulphide water, and dissolved in nitric acid (1:1). After filtration, the filtrate is concentrated with 10 c.c. of sulphuric acid (1:5) till the nitric acid has been expelled. The residue is diluted to 100 c.c. with water and allowed to stand for some hours, to complete the precipitation of lead sulphate.

Lead.—Jamieson recommends weighing the lead sulphate on a Gooch crucible. This, however, is rarely possible. But reliable results are obtained by dissolving the lead sulphate in ammonium or sodium acetate solution and estimating the lead colorimetrically. Under the conditions of the experiment small quantities of lead are precipitated practically quantitatively, as the following experiments prove.

Definite volumes of a standard solution of lead nitrate were treated with sulphuric acid. In one series the nitric acid was removed by concentration, whilst in another it was left in the solution. The precipitated lead sulphate was collected on a Gooch filter and weighed, and any lead in the filtrates was estimated by the colorimetric method. (The presence of alcohol does not interfere with the colorimetric determination of lead.) The results obtained indicate that, even with very small quantities of lead, 99 per cent. is precipitated as lead sulphate when the usual procedure is followed, whilst with incomplete removal of nitric acid, 95 per cent. is obtained as lead sulphate. The figures are given in the following table:

Exp.	Vol. of strong Lead sol. taken. cc.	Lead taken. Grm.	Weight of $PbSO_4$ Grm.	Lead. Grm.	Lead in filtrate Grm.	Total Lead recovered Grm.	Lead pptd. as $PbSO_4$. Per Cent. (by diff.)	Lead pptd. as $PbSO_4$. Per Cent. (from Column 4).
I.								
1	20	0·0200	0·0252	0·0178	0·0014	0·0192	93·0	89·0
2	10	0·0100	0·0134	0·0092	0·0006	0·0098	94·0	92·0
3	7·5	0·0075	0·0104	0·0071	0·0004	0·0075	95·3	94·6
4	5	0·0050	0·0070	0·0048	0·0002	0·0050	95·4	96·0
II.								
1	20	0·0200	0·0275	0·0188	0·0002	0·0190	99·3	94·0
2	10	0·0100	0·0138	0·0094	0·0001	0·0095	99·0	94·0
3	5	0·0050	0·0072	0·0049	0·0002	0·0051	96·4	98·0

The lead is prepared for tinting by solution of the sulphate in hot sodium or ammonium acetate (1:3), in which the lead sulphate dissolves unchanged. The asbestos pad containing the lead sulphate is digested with 15 to 20 c.c. of the hot acetate solution, the liquid made up to 50 or 100 c.c., and the lead estimated colorimetrically in an aliquot part. The following results indicate that 2 extractions of the asbestos are necessary.

		Lead. Grm.
I.	1st extraction with 15 c.c. ammonium acetate (1:3) gave	0·0005
	2nd in similar way .. .. .. ,,	0·0002
	3rd ,, ,, ,, ... ,,	0·00003
II.	1st ,, ,, ,, ... ,,	0·0006
	2nd ,, ,, ,, ... ,,	0·0002
	3rd ,, ,, ,, ... ,,	0·0000

It is, of course, necessary to ascertain that the acetate solution used is free from lead. One experiment indicated that extraction with sodium acetate was more efficient than with ammonium acetate, but this result was not confirmed.

It is also advisable to add an equivalent amount of ammonium (or sodium) acetate solution to the cylinder containing the standard lead solution, so that the comparison is made with both solutions containing the same amount of acetate. Finally, in the procedure recommended by Jamieson (see above) a small amount of lead will be lost when the metallic sulphides are dissolved in hot nitric acid. It has been found that, even with such small quantities of lead sulphide, solution in nitric acid results in the formation of some lead sulphate. It is therefore necessary to estimate this by extraction of the asbestos pad with ammonium acetate solution. The lead is estimated in this solution colorimetrically, and the amount found is added to the main bulk of the lead estimated as described above.

Copper.—Copper is precipitated as sulphide. Jamieson collects the copper sulphide in a Gooch crucible, ignites it and weighs the oxide. This, as with lead, is very unreliable. In a good gelatin the error of experiment would be greater than the copper present. The following method gives accurate results:—The copper sulphide is dissolved in nitric acid, the solution evaporated to dryness, the residue re-dissolved, and the solution electrolysed. The copper is then dissolved in nitric acid, and the solution is evaporated to dryness. The copper nitrate is dissolved in water, the solution made up to 100 c.c. and the copper estimated colormetrically with potassium ferrocyanide. Winkler, *J. Soc. Chem. Ind.*, 1913, **32**, 157, states that, in the absence of lead, copper in water is best estimated colorimetrically as sulphide, with the addition of ammonium chloride, which increases the intensity of the coloration. This has been tried, but is not so satisfactory as the ferrocyanide method. Wynter Blyth recommended the addition of ammonium nitrate solution to the Nessler cylinders. This overcomes the difficulty that a redder shade is given by the separated copper than by the standard solution. Ammonium acetate acts in the same way, and ensures the absence of traces of mineral acid. It is important to add the ammonium nitrate or acetate solution before the ferrocyanide. If added afterwards, no effect is produced. Excess does not affect the colour. From 2 to 3 c.c. of a 10 per cent. solution is a suitable quantity.

Attempts were made to estimate the lead and copper together, colorimetrically, by means of sodium sulphide, first with, and then without, the addition of potassium cyanide. The method was found to be quite unreliable. Thresh (ANALYST, 1924, 124) has arrived also at the same conclusion in the case of water. But good results were given by the following process which avoids electrolysis of the copper:

The mixed sulphides, obtained as described above, are dissolved in nitric acid, and the solution is evaporated to dryness. The nitrates are dissolved in 100 c.c. of water containing 3 c.c. of hydrochloric acid, and the solution treated with hydrogen sulphide. The amount of acid used is of importance. If the solution is not sufficiently acid, zinc sulphide is precipitated, whilst too much acid involves incomplete precipitation of lead. The sulphides of lead and copper are dissolved in nitric acid, the solution is evaporated to dryness, and the nitrates dissolved in water. Lead is estimated in one portion of the solution in the usual manner, and the copper in another by means of potassium ferrocyanide in the presence of ammonium acetate.

Bogue and Hertwig both recommend the iodimetric estimation of copper in gelatin. The copper nitrate, obtained as described above, is dissolved in water, 2 grms. of potassium iodide are added, and the liberated iodine is titrated with 0·01 *N* sodium thiosulphate solution (1 c.c. =0·000636 grm. Cu). The process has been tested, but it was found that the quantity of copper present in good gelatins is rarely sufficient for accurate results. If a gelatin contained 30 parts of copper per million and 30 grms. were taken for analysis, the volume of thiosulphate used would be only 1·4 c.c.

Zinc.—The method of Jamieson is adopted. The filtrate from the copper sulphide is boiled to expel hydrogen sulphide, and made alkaline with ammonia. Excess of formic acid is then added and the zinc precipitated with hydrogen sulphide, collected in a Gooch crucible, ignited and weighed as oxide. If iron oxide and alumina are precipitated when ammonia is added, they are removed by filtration. But this is not necessary, since the sulphides of both iron and aluminium are soluble in formic acid.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE COMPOSITION OF COCONUT OIL.

IN a recent number of the *Journal of the Chemical Society* (*J. Chem. Soc.*, **1923**, **123**, **2837**), E. V. Walker gives the results of the examination of a sample of coconut oil, and refers particularly to the proportion of octoic and decoic acids, commonly termed caprylic and capric acids respectively. This author finds about 6 per cent. of caprylic and from 1 to 2 per cent. of capric acid, and compares these figures with those of Paulmyer, who found 0·25 per cent. of caprylic and 19·5 per cent. of capric acid, and with those of the writer (Elsdon, ANALYST, 1913, 38, 8), who found 9 per cent. of caprylic and 10 per cent. of capric acid. Walker states that "This (his analysis) is entirely at variance with the analyses quoted in the first part of the paper, but is in agreement with Georgey's observations. Whether this disagreement represents real differences in the composition of different samples of oil or whether the analyses were inaccurate cannot be stated definitely."

In regard to the analyses of the writer, Walker entirely overlooks the fact that it was clearly stated in the paper quoted, and also in a later one (ANALYST, 1914, **39**, 78), that the method used had little quantitative significance, although a fairly obvious misprint occurs in the last line but one of the second paper, where "qualitative" should read "quantitative." The method adopted, that of alcoholysis, is now well known. The figures given in the papers by the writer were obtained by separating the mixed methyl esters into fractions by distillation, the receiver being changed at a point half way between the boiling points of consecutive esters. In the case of coconut oil the fraction corresponding to methyl laurate was 442 grms., whereas that corresponding to methyl caprate was 92·5 grms., and it is, of course, perfectly obvious that in a fractionation of this type the large quantity of methyl laurate present in the mixed esters will increase considerably the next lower fraction to it, which is methyl caprate. It is thus very probable, and a careful reading of the paper would have indicated this quite clearly, that the amount of capric acid actually present in coconut oil is considerably less than the 10 per cent. apparently indicated by the weight of the methyl caprate fraction.

The writer has more recently separated the fatty acids from about 10 kilos. of coconut oil, and from the amount of capric acid actually isolated from this he is quite in agreement with Walker that there is considerably less than 10 per cent. of capric acid present in coconut oil, and that probably the figure of from 1 to 2 per cent. is not very far from the truth. It is possible that the method of Crowther and Hynd (*Biochem. J.*, 1917, **11**, 139) would settle this matter definitely. These workers have obtained apparently excellent results, from an extention of the original method, in their examination of butter fat, and it seems quite likely that their modifications will greatly increase the quantitative accuracy of the process.

G. D. ELSDON.

SENSITIVE REACTIONS FOR COPPER.

ON adding to a dilute solution of a copper salt in water three or four drops of a dilute solution of ammonium or potassium thiocyanate, and then a few drops of a one per cent. solution of benzidine in alcohol, a deep blue precipitate, insoluble in all the usual organic solvents, is obtained; this reaction is sensitive for one part of copper in a million and for one part of thiocyanogen in 13000.

If a half per cent. alcoholic solution of guaiacum resin be substituted for the benzidine in the above reaction a blue colour is developed with a concentration of one part of copper in ten millions.

More delicate still is the following test:—If a few drops of a half per cent. solution of guaiacum resin in alcohol be added to a small quantity of a weak aqueous solution of a copper salt and the mixture heated, a deep blue colour develops, the blue compound being soluble in chloroform. This reaction is sensitive for one part of copper in two hundred millions. The above reactions are not given by any of the other metals. It is true that iron gives a blue colour with guaiacum in strong solutions, but this colour disappears on heating.

ROBERT FLEMING.

THE SCHOOL OF MEDICINE,
CAIRO.

CONDENSED MILK CALCULATIONS.

The Condensed Milk Regulations, 1923, required the label on a tin to declare the equivalent of the contents in pints of milk or skimmed milk. The standard milk must contain at least 12·4 per cent. of milk solids and at least 3·6 per cent. of milk fat, and skimmed milk at least 9 per cent. of milk solids-not-fat.

The following table gives the number of ounces of constituents in the usual quantities of milk given on condensed milk declarations. The third decimal place is ignored. Milk is presumed to have a specific gravity of 1·032; and machine-skimmed milk 1·035, on the basis of 8·9 per cent. of solids-not-fat and 0·1 per cent. of fat.

Pints.	¾	⅞	1	1½	1⅝	1¾	1⅞	2
Milk (full cream).								
Milk solids, ozs.	1·91	2·23	2·55	3·83	4·15	4·47	4·79	5·11
Milk fat, ozs.	0·55	0·64	0·74	1·11	1·20	1·30	1·39	1·48
Machine skimmed milk.								
Milk solids-not-fat, ozs.	1·39	1·63	1·86	2·79	3·02	3·26	3·49	3·73

For example, a condensed milk contained 34·8 per cent. of milk solids and 9·8 per cent. of milk fat and was labelled to be equivalent to 1¾ pints of milk; the weight of the contents was 13·8 ozs. It therefore contained 4·80 ozs. of milk solids and 1·35 ozs. of milk fat, and was in excess of the requirements of 4·47 ozs. and 1·30 ozs., respectively, shown in the above table.

The Regulations make no distinction in the composition of machine-skimmed milk and skimmed milk, though Section 7 suggests that a distinction in the labels should be made.

When the label gives directions for dilution, the fluid produced must be equal to the above standard milk unless a clear statement is made to the contrary; the calculation is as follows:

Percentage in diluted milk $= \frac{S \times P}{S+V}$, where S is specific gravity of the condensed milk, P is the percentage composition of a given constituent, and V is the volume of water directed to be added to one volume of milk.

J. F. Liverseege.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

LABELLING OF MARGARINE.

Hawes *v.* Stephens.

On May 1st an appeal from a decision of the Clerkenwell magistrate (Mr. Bingley) was heard in the High Court by the Lord Chief Justice and Justices Shearman and Roche.

The appellant, a grocer, had been summoned for selling margarine described by a name other than "margarine," or a name combining the word "margarine" with a fancy or other descriptive name approved by the Board of Agriculture and

Fisheries, contrary to Sec. 8 of the Butter and Margarine Act. He had been convicted and fined 20s.

The respondent (a food inspector) had asked to be supplied with "Kernut" margarine, and had been supplied with an article wrapped in two wrappers, the outside one having on it the word "margarine" in half-inch letters, and the inner one the words "Kernut margarine," printed in capitals, and beneath it in smaller type "churned with fresh milk."

The Public Analyst for Islington had certified that the sample contained 100 per cent. of margarine as defined by Sec. 13 of the Act of 1907.

On behalf of the respondent it had been contended before the magistrate that the appellant was limited to the use of the approved word "Kernut," and that the additional words "churned with fresh milk" formed part of the descriptive name, and were not permitted under Sec. 8 of the Act. The case for the appellant had been that the words "churned with milk" did not form part of the descriptive name, but were only an intimation to purchasers of the method used in the preparation of the article.

Mr. Barrington Ward, on behalf of the appellant, contended that the magistrate had been wrong in holding, on the authority of the Scottish case of *Patterson* v. *Maypole Dairy Co.*, that the words in question were part of the descriptive name, and that it had not been suggested that the appellant had not complied with the law so far as concerned the outside wrapper.

Mr. Burrows, for the respondent, urged that the added words on the inner wrapper were part of a descriptive name, and that they suggested to the purchaser that he was getting margarine "churned with fresh butter"—not that he was getting Kernut margarine. In his submission this case was indistinguishable from the Scottish case.

In giving the judgment of the Court, the Lord Chief Justice said that the only question was whether upon the facts stated the magistrate had been right in his decision in considering himself bound by the Scottish case of *Patterson* v. *Maypole Dairy Co.* In that case the words used were "Mayco Margarine mixed with Maypole butter," whereas in the present case quite different words were used. Each case depended upon the particular words used. It was because the magistrate thought that the two cases were indistinguishable that he came to the conclusion he did. If those were the grounds, there could be no doubt that he had made a mistake in a point of law, and that he ought to have dismissed the summons, inasmuch as the added words were not part of the fancy or descriptive name.

The other judges concurred, and the Court allowed the appeal, with costs.

SWEET SPIRIT OF NITRE DEFICIENT IN ETHYL NITRITE.

On April 24th a pharmacist was summoned at Cheltenham for the sale of sweet spirit of nitre deficient in ethyl nitrite to the extent of at least 45 per cent.

Evidence was given by the inspector who made the purchase that the defendant had explained to him that this sweet nitre was part of the stock which he had taken over with the business two years previously, and that he could not throw it away.

For the defence it was urged that the analyst's certificate was bad on two grounds: (1) Sweet nitre was peculiarly liable to decomposition, and the certificate did not state, as it should, whether or not any change had taken place in the constitution of the article that would interfere with the analysis. (2) The certificate was too vague, and did not contain sufficient data to enable the magistrates to form an opinion for themselves.

As evidence that the article was prone to decomposition, attention was directed to the wide range in strength permitted by the B.P., viz. from 2·66 to 1·52 per cent. The fact that the spirit was ordered to be kept in closed vessels, as had been done in this case by the defendant, also pointed in the same direction. Extracts from Martindale's Extra Pharmacopœia were quoted to show that the decomposition of sweet spirit of nitre would occur even in closed vessels.

The Chairman of the Bench here observed that they were satisfied as to the decomposition, but considered that two years was too long to keep such a substance.

Mr. Haddock (for the defence) then referred at length to the vinegar of squill case of *Hudson* v. *Bridge*, in which it was held by the High Court that the form prescribed by the Act must be adhered to in the case of a decomposable article.

Evidence was also given by a retired pharmacist that sweet nitre would undergo decomposition under the most "favourable conditions," and that it had been proved that the decomposition was due to the presence of water. He agreed with "Squire's Companion to the British Pharmacopœia" that, even in closed vessels, a loss of as much as 6 per cent. per month was possible.

The Bench dismissed the summons on technical grounds, but considered that the case was not at all satisfactory.

Connecticut Agricultural Experiment Station.

TWENTY-SEVENTH REPORT ON FOOD PRODUCTS (1922).

STATISTICS are given of the 2546 samples of food and drugs examined during the year, together with details of their analysis and composition. Among the points of more than local interest dealt with reference may be made to the following:

SACCHARIN IN CARBONATED BEVERAGES.—In 1920 a State law was passed regulating the manufacture and bottling of non-alcoholic beverages, and prohibiting the use of saccharin. In 1920, 40·5 per cent. of the samples examined contained saccharin; in 1921 the percentage had fallen to 18·7 per cent.; and in 1922 to 5·4 per cent.

DIABETIC AND SPECIAL FOODS.—A sample of *casein flour* had the following composition:—Moisture, 7·44; ash, 3·49; protein ($N \times 6{\cdot}38$), 56·72; lactose, 7·52; and fat, 19·76 per cent.

Washed gluten flour contained:—Moisture, 7·10; nitrogen, 14·05; protein ($N \times 5{\cdot}7$), 80·09; soluble carbohydrates (as dextrose), 0·55; and starch, 2·81 per cent.

A *whey mixture* contained:—Total solids, 16·49; ash, 0·76; protein ($N \times 6{\cdot}38$), 6·25; lactose, 0·55; fat, 8·56; and gelatin, 0·37 per cent.

The question was raised by a manufacturer of diabetic food products as to the possible loss of fat in foods during the baking process. A series of trials gave results showing no significant difference in composition between doughs prepared from the flours and the corresponding baked products.

COMMERCIAL INULIN.—The sample contained:—Moisture, 9·53; nitrogen, 0·16; ash, 2·58; insoluble in water, trace; reducing sugar (as lævulose), 0·79 per cent.; monosaccharide, present. Specific rotation at 20° C., corrected for moisture, ash and protein, −32·9°. The specific rotation of inulin prepared from dahlia tubers in the laboratory was −36·5 (corr.).

EGGS.—Of 48 samples of eggs retailed as *fresh* eggs, 3 were classified as fresh, 45 as not fresh, but edible, and 1 was inedible.

The characteristics of fresh eggs and eggs which are not fresh are thus defined:*

CHARACTERISTICS OF A FRESH EGG.

Before the candle.

Air space: Not enlarged; less than three-fourths inch in diameter.

White: Firm and clear.

Yolk: Dimly seen through the white as a shadowy object indistinct in outline. The chick spot is not visible.

Distinguishing characteristics: No shrinkage and general firm conditions of white and yolk. Edible.

Out of shell.

White: Firm and thick; opalescent; reflects the light.

Yolk: Spherical and firm; chick spot small with no sign of hatching. Color is uniform for the entire yolk, but varies in color from light yellow to deep orange, and is occasionally olive green.

Distinguishing characteristics: General firm condition of white and yolk. White, opalescent.

CHARACTERISTICS OF AN EGG WHICH IS NOT FRESH.

Before the candle.

Air space: Enlarged; the lower wall may be movable in outline.

White: Thin and clear.

Yolk: Definite in outline; sometimes weak, and may occasionally have dark mottled areas.

Distinguishing characteristics: Enlarged air cell and increased contrast between white and yolk as compared with a fresh egg. Edible.

Out of shell.

White: Thin, no opalescence, does not reflect the light as much as does a fresh egg.

Yolk: Flattened, and occasionally may have light, mottled areas.

Distinguishing characteristics: Thin white and flattened yolk.

Laws regulating the distribution and sale of eggs aim to insure that the consumer obtains good, edible eggs always, and fresh eggs if the extra price of such is paid. If, however, the consumer's understanding of a fresh egg be one that is but two or three days old, then he seldom gets what he expects. If he accepts fresh eggs to be those possessing the characteristics here defined for fresh eggs, he obtains such eggs much more frequently. Whatever his idea of fresh may be, it can be positively stated that the elapsed time since an egg was laid is not the determining factor in establishing its freshness; the conditions of storage are all-important. As to how old an egg may be and still retain the characteristics of a fresh egg, it is pertinent to quote the following:†

> "An egg laid in March or April and kept under proper conditions will retain the characteristics which distinguish a fresh egg for from three to four weeks. In warmer weather this time would necessarily have to be reduced, and an egg laid in very hot weather and possibly allowed to remain in the nest for twenty-four hours or more, has lost these characteristics to such an extent that it is not as good as an April egg kept for a month under favourable conditions, and it should not be offered for sale nor be permitted to be sold as and for a fresh egg.
>
> "Nor can an egg which is allowed to remain exposed to ordinary atmospheric conditions in a retail store for several days or a week in warm weather be expected to retain the characteristics which are expected of a fresh egg."

VANILLA EXTRACT.—This is defined‡ as the flavouring extract prepared from the vanilla bean, with or without sugar or glycerin, and containing, in 100 c.c., the soluble matter from not less than 10 grms. of the vanilla bean. Such extracts have been found to contain from 0·11 to 0·31 per cent. of vanillin. In 17 commercial samples examined the proportion of vanillin ranged from 0·09 to 0·26 per cent.

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* *U.S. Dept. Agr. Bull.,* 565, p. 13 (1918).

† *Penn. Dept. Agr., Bureau of Foods,* 17, p. 44 (1919).

‡ U.S. Dept. Agric. Circ. 136.

VANILLA SUBSTITUTES.—Two samples contained high percentages (0·57 and 0·67) of vanillin, probably synthetic, together with 0·08 and 0·06 per cent., respectively, of coumarin.

MEAT PRODUCTS.—Five of eight samples of Hamburg steak were illegally preserved with sulphites, and three were passed. State regulation 7 provides that no exception will be taken to foods which contain ordinary amounts of sulphur dioxide, provided the fact is declared. This is intended to exempt certain products such as molasses, dried fruits and wines which have been subjected directly or indirectly to the process of sulphuring. The use of sulphites to preserve the fresh appearance of, or deodorise meat is not contemplated by this regulation. The five samples condemned contained sulphurous acid in amounts ranging from 260 to 3300 mgrms. per kilo.

CRYOSCOPY OF MILK.—Results, cited in detail, show that the effect of the increased acidity of milk upon freezing-point depression is an additive factor, and that the magnitude of the increased depression closely approximates 0·003° C. for each 0·01 per cent. increase in acidity. Such correction for acidity ought to be made in the observed freezing point depression in the case of milk which is sensibly sour. It would seem that, in general, acidities in excess of 0·20 to 0·25 per cent. result from fermentation, and will represent milk which is sour or near the "turning" point. No correction of freezing point is recommended for acidity within the range normal for fresh milk, *i.e.* milk showing an acidity not exceeding 0·20 per cent.

The freezing points of milk from tubercular cows, or cows otherwise abnormal physically, have again, in general, been found within the limits for normal milk, the few exceptions noted being in the direction of decreased depressions.

The value of the cryoscopic method as an adjunct to other methods of detecting water has been fully demonstrated by data covering a period of more than 2 years. Its use is unnecessary when present methods furnish conclusive evidence, but, in the opinion of the majority of experienced workers, its unique value is shown in those cases where the evidence of present methods is conflicting or inconclusive.

HUMAN MILK.—Analyses of 21 samples are given. The fat content ranges from 1·2 to 6·4 per cent., but it is pointed out that the amount of fat varies widely in different portions of the milk, and that unless the entire secretion of the gland is drawn and well mixed before sampling the result obtained may be very misleading.

International Union of Pure and Applied Chemistry.

SUGGESTED STANDARDS FOR HYDROCHLORIC ACID, SODIUM CHLORIDE AND ZINC.

THE British Committee (Messrs. Carr, Fox, Hinks, and Marlow) have sent the following report on Standards for Hydrochloric Acid, Sodium Chloride and Zinc to Mr. Carr, the convener, who will put them before the Union Internationale.

ACID HYDROCHLORIC.

APPEARANCE AND COLOUR.—The acid shall be clear and colourless.

DETERMINATION.—The acid shall contain 35 per cent. by weight of hydrogen chloride. This is to be determined by adding 3 or 4 grms. of the acid to 50 c.c. of water and titrating with 0·1 *N* sodium hydroxide, methyl red being used as indicator.

Tolerance.—Acid containing between 34 per cent. and 36 per cent. by weight of hydrogen chloride may be accepted as conforming to this standard.

Residue on Evaporation and Ignition.—Fifty c.c. of the acid shall be evaporated to dryness in a platinum basin on a water bath and the residue ignited at 600° C., and weighed. The residue shall not exceed 0·0005 grms.

Sulphuric Acid.—Fifty c.c. of the acid shall be evaporated till the bulk is reduced to about 5 c.c. The residue is diluted to 50 c.c., heated to boiling, and 5 c.c. of 0·1 *N* barium chloride solution is then added. No precipitate should be formed after setting aside for 24 hours.

Heavy Metals.—Ten c.c. of the acid shall be diluted with 50 c.c. of water and mixed with 50 c.c. of a freshly prepared solution of hydrogen sulphide. No darkening in colour should result when compared against a mixture of equal parts of distilled water and hydrogen sulphide solution when viewed in a Nessler glass through a depth of 10 cm.

Free Chlorine.—Five c.c. of acid are diluted with 45 c.c. of water, followed by the addition of half a c.c. of one per cent. solution of zinc iodide and 2 c.c. of starch solution. The liquid is then stirred; no blue coloration shall develop within 5 minutes.

Iron.—Five c.c. of the acid shall be diluted with 50 c.c. of water, and 2 c.c. of freshly prepared solution of potassium ferrocyanide shall be added. The colour shown in 30 minutes shall not be greater than that produced by 10 c.c. of a solution of ferric chloride containing 0·0001 grm. of iron per 100 c.c.

Arsenic.—The arsenic shall not exceed 0·04 part per million calculated as arsenious oxide (As_2O_3). The test shall be carried out as follows:—To 50 c.c. of the acid add 0·5 c.c. of bromine solution (arsenic-free) and evaporate on a water bath until the volume is reduced to about 15 c.c. Add 50 c.c. of hot water followed by 0·5 c.c. of stannous chloride solution (arsenic-free) and apply the Marsh-Berzelius or Gutzeit test to the liquid for 30 minutes.

Sulphites.—To 50 c.c. of water add 0·05 c.c. of 0·1 *N* iodine solution and 2 c.c. of starch solution. Dilute 5 c.c. of the acid with 50 c.c. of water and add it to the iodine-starch solution. The blue colour shall persist after mixing.

SODIUM CHLORIDE.

A white, crystalline salt; a solution of 5 grms. in 50 c.c. of water should be colourless and neutral, and should not show any turbidity when viewed in a Nessler glass through a depth of 10 cm.

Determination.—Five grms. should not lose more than 5 mgrms. on drying at 100° C. to constant weight; 0·2 grm. dissolved in 100 c.c. of water, a few drops of potassium chromate solution added, and the solution titrated with 0·1 *N* silver nitrate solution, should show it to contain at least 99·9 per cent. of sodium chloride.

Sulphate.—Five grms. dissolved in 50 c.c. of water, with addition of 1 c.c. of hydrochloric acid, and heated to boiling; 2 c.c. of barium chloride solution added should not give any turbidity on allowing the mixture to stand for 24 hours.

Bromide.—Two grms. of the salt shall be dissolved in 10 c.c. of water in a porcelain dish, and 0·1 c.c. of chlorine water, prepared by diluting 1 volume of saturated chlorine water with 4 volumes of water, shall be added. No yellow coloration should be produced.

Iodide.—To a solution of 5 grms. of the salt in 20 c.c. of water, add one drop of ferric chloride solution and 1 c.c. of starch solution. There must not be any blue coloration produced.

Ammonia.—Two grms. dissolved in 20 c.c. of water, 1 c.c. of Nessler's solution added. There should be no greater change than that produced by the addition of 1 c.c. of Nessler's solution to 20 c.c. of the same water with which the solution of the sample was effected.

Potassium.—Dissolve 1 grm. in 10 c.c. of water, add 5 c.c. of sodium cobaltinitrite solution, allow the mixture to stand for 24 hours. No precipitate should be formed.

Alkaline Earths.—Two grms. dissolved in 20 c.c. of water should not show any turbidity on adding dilute sulphuric acid and standing two hours. Two grms. dissolved in 20 c.c. of water should not show any turbidity on adding a few drops of 5 *N* ammonia, 1 c.c. of 0·1 *N* ammonium oxalate and 1 c.c. of 0·1 *N* ammonium phosphate solution, and allowing it to stand for 24 hours.

Iron.—Five grms. shall be dissolved in 50 c.c. of water, and 2 c.c. freshly prepared solution of potassium ferrocyanide shall be added. The colour shown in 30 minutes shall not be greater than that produced by 10 c.c. of a solution of ferric chloride containing 0·0001 grm. of iron per 100 c.c.

Heavy metals.—Ten grms. shall be diluted with 50 c.c. of water and mixed with 50 c.c. of a freshly prepared solution of hydrogen sulphide. No darkening in colour should result when compared against a mixture of equal parts of distilled water and hydrogen sulphied solution when viewed in a Nessler glass through a depth of 10 cm.

ZINC.

1. **Arsenic.**—When the Gutzeit or Marsh-Berzelius test is applied to 10 grms., using arsenic-free acid, no stain or mirror shall be obtained in 30 minutes.

2. **Sensitiveness and Activity.**—When tested in the Gutzeit or Marsh-Berzelius apparatus, using arsenic-free acid, with the addition of 0·002 mgrm. of arsenious oxide (As_2O_3), a visible stain or mirror of normal intensity should be obtained. A steady stream of hydrogen should be evolved.

3. **Impurities Oxidisable by Potassium Permanganate.**—Ten grms. are dissolved in a mixture of 50 c.c. of water and 15 c.c. of concentrated sulphuric acid in a closed vessel fitted with a valve; the zinc should be entirely dissolved, leaving no black flakes. As soon as solution is complete, 0·1 *N* potassium permanganate solution is added; 0·1 c.c. of this solution should produce a permanent pink colour.

4. **Sulphur, Phosphorus, etc.**—One grm. of zinc and 2 c.c. of concentrated arsenic-free sulphuric acid diluted to 10 c.c. with water are placed in a narrow test tube containing a plug of cotton wool in its upper part. Over the mouth of the tube is placed a disc of filter paper which has been moistened with aqueous silver nitrate solution (1:1) and dried. After the reaction has proceeded for 2 hours in the dark and in an atmosphere free from sulphuretted hydrogen, the paper should show no yellow or black stain.

Note.—The zinc tests for arsenic testing must comply with tests 1, 2 and 4, and zinc for reducing purposes must comply with tests 3 and 4.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Identification of Wheat and Rye Flours by means of their Aqueous Extract. R. Strohecker. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 90–97.)—Aqueous extracts of wheat and rye flours have characteristic and differing refractive indices and electrical conductivities; these factors are available for their recognition and estimation in mixtures. Five grms. of the flour are made into a thin paste with 50 c.c. of distilled water,* triturated for 15 minutes and then filtered; 10 c.c. of the bright filtrate are evaporated to dryness and weighed. The refractive index of the solution is determined by an immersion refractometer at 17·5° C. and the electrical conductivity at 18° C. Examination of a large number of samples shows that the following are the limits, calculated on the dry sample, for wheat and rye meals:

	Wheat	Rye.
Water extract, per cent.	3·83– 8·58	11·80–17·08
Refraction (Zeiss) ,,	18·75–20·96	22·15–24·27
Conductivity $\times 10^{-4}$,,	5·24–10·24	10·45–14·33

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* Conductivity water not specified.

The conductivity, being mainly due to salts, is closely related to the ash content, but is more readily determined. The quantities of each meal in a mixture of the two can be calculated approximately from the following table:

	Water Extract.	Refraction.	Conductivity.
Wheat flour (85 per cent. extraction)	7·31	20·11	8·52
90 per cent. wheat, 10 per cent. rye	7·54	20·68	8·89
75 ,, ,, 25 ,, ,,	8·94	20·90	9·17
50 ,, ,, 50 ,, ,,	10·87	21·79	9·99
25 ,, ,, 75 ,, ,,	12·94	22·73	10·56
10 ,, ,, 90 ,, ,,	13·64	22·90	11·26
Rye flour (85 per cent. extraction)	14·75	23·33	11·38

H. E. C.

Effect of Sodium Trichloroacetate on Sugar Estimations. D. Stiven. (*Biochem. J.*, 1924, **18**, 19–21.)—When trichloracetic acid was used to precipitate proteins and the glucose in the filtrate was estimated either by the Bertrand method or the micro-method of Schaffer and Hartmann (*J. Biol. Chem.*, 1921, **45**, 377) it was observed that the percentage of glucose found was lower than in the case of control tests on a solution not treated with trichloracetic acid. This fact was traced to the presence of sodium trichloracetate which, in sufficiently large quantities, can inhibit the reduction of the copper solution. Therefore the use of trichloracetic acid, as a protein precipitant prior to glucose estimations by any of the copper reduction methods, is to be avoided. Experiments are described on the Schaffer-Hartmann micro-method, which show the extent of the inhibiting effect and also afford evidence of a reaction between the copper of the Schaffer solution and the sodium trichloracetate by which the copper passes into a non-reducible complex. This organo-copper compound decomposes gradually on heating; but during the heating the glucose is destroyed by the sodium carbonate of the copper solution and no reduction (or very little) results, even after the copper has reverted to its reducible condition. Polarimetric evidence of a reaction between the glucose and trichloracetic acid is entirely negative. The addition of the trichloracetate at any stage stops the reduction, and the cuprous oxide already formed is not destroyed. P. H. P.

Glycerides of Palm-kernel Oil. A. Bömer and K. Schneider. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 61–89.)—The glycerides of oil extracted from Porto Novo palm kernels were separated by more than 100 fractional crystallisations from acetone and divided into five principal fractions having m.p. 13·9° to 51·4° C. After determination of the usual constants, the fatty acids of these fractions were prepared and examined by well-known methods. Contrary to the results of Elsdon (ANALYST, 1914, **39**, 455) and others, no caproic or stearic acid was present, and the presence of capric acid was doubtful. The fatty acids found were caprylic, lauric, myristic, palmitic and oleic, and the glycerides caprylo-myristo-olein, myristo-dilaurin and lauro-dimyristin, together with small quantities of palmito-dimyristin and myristo-dipalmitin. Comparison with the results obtained for coconut oil (Bömer and Baumann, *Zeitsch. Unters. Nahr. Genussm.*, **1920, 40, 97**) shows these oils to contain the following glycerides, No. 1 predominating in quantity:

	Coconut Oil.		Palm-kernel Oil.	
I.	Caprylo-lauromyristin	m.p. 15° C.	Caprylo-myristo-olein	m.p. 13·9° C.
II.	Myristo-dilaurin	,, 33°	Myristo-dilaurin	,, 33·4°
III.	Lauro-dimyristin	,, 38·1°	Lauro-dimyristin	,, 40·0°
IV.	Palmito-dimyristin	,, 45·1°	Palmito-dimyristin	,, 45·2°
V.	Stearo-dipalmitin	,, 55°	Myristo-dipalmitin	,, 51·4°

H. E. C.

Detection of Glycerides in Beeswax. P. Bourcet. (*Bull. Soc. Chim.*, **1924, 35,** 421.)—The author's application of Denigès' test for methyl glyoxal to the detection of glycerides in beeswax is as follows:—Two to 3 grms. of yellow beeswax are saponified with 10 to 15 c.c. of potassium hydroxide solution strength in a large test tube for 1 hour, with frequent shaking; 15 c.c. of water are added, and the solution slightly acidified with 25 per cent. sulphuric acid and filtered. Two c.c. of the filtrate are mixed with 20 c.c. of saturated bromine water, and boiled on a water bath for 20 minutes, when the mixture may be decolorised; but, if not, the boiling and shaking are continued till the colour has disappeared. A tenth of a c.c. of 0·05 per cent. alcoholic solution of codeine and 2 c.c. of the brominated liquid are put into a test tube and 5 c.c. of pure sulphuric acid (sp. gr. 1·84) added, the mixture shaken, and the tube placed in a boiling water bath for 2 minutes, when, if glycerides are present, a bluish-green coloration will be observed. Since an examination of a very large number of authenticated samples of yellow beeswax from various sources failed to give a positive reaction, the coloration can be taken to indicate the presence of added glyceride, and is sufficiently sensitive for all ordinary analytical purposes. The coloration is sometimes obtained with genuine bleached beeswax. D. G. H.

Use of Ethylene in the Colouring of Citrus Fruits. E. M. Chace and F. E. Denny. (*Ind. Eng. Chem.*, **1924, 16,** 339–340.)—For a long time it has been the practice to colour citrus fruits by exposing them to the products of combustion of oil. Ethylene has been proved to be the gaseous constituent which changes the colour of the pigment from green to yellow, and pure ethylene is now used for the purpose. It is stated that one part by volume of ethylene in one million parts of air causes the fruit to change colour in about the time required by the older stove method; the colour change is more rapid when the concentration of the ethylene is one part in 5000 to 20,000 parts of air. These mixtures are the more convenient to use and are non-explosive. W. P. S.

The Solanine Content of Potatoes. A. Bömer and H. Mattis. (*Zeitsch. Unters. Nahr. Genussm.*, **1924, 47,** 97–127.)—After examination of known methods for the estimation of solanine in potatoes the following, more accurate, process was devised: From 200 to 300 grms. of the finely-divided potatoes are mixed with 250 c.c. of water, extracted for 30 minutes at room temperature, and the liquor pressed out. The mass is re-extracted three times with 250 to 300 c.c. of water containing 0·5 c.c. of acetic acid. The mixed extract is made alkaline with ammonia, and, after addition of 10 grms. of kieselguhr, evaporated to dryness on the water bath, finely powdered, and extracted with boiling 95 per cent. alcohol for 6 hours. The alcoholic solution is evaporated, the residue dissolved in 50 to 100 c.c. of water acetified with acetic acid, and the solution made alkaline with ammonia and warmed for 30 minutes to coagulate the solanine precipitate, which is then filtered off and washed with a 2·5 per cent. solution of ammonia. The crude solanine so obtained is dissolved in very dilute acetic acid, re-precipitated with ammonia, washed, dried at 100° C. and weighed. The second precipitate should be almost colourless. A correction of 2·75 mgrms. for each 100 c.c. of solution, including wash liquor, is applied, on account of the solubility of the base in dilute ammonia.

The normal solanine content of potatoes depends upon the variety; usually it is from 2 to 10 mgrms. per 100 grms.; those kinds in which the amount exceeds 25 mgrms. are injurious, and are more harmful when cooked in their skins than when previouly peeled. Unripe potatoes contain more solanine than the same variety when ripe and smaller ones more than large ones. Prolonged exposure to daylight causes an increase in the solanine content, but no connection is apparent between the quantity of this base and the fertiliser applied to the soil. H. E. C.

True Nature of the Methyl Salicylate Glucoside in the Bark of the Sweet Birch. M. Bridel. (*Comptes rend.*, 1924, **178**, 1310–1312.)—The glucoside gaultherin extracted from the bark of *Betula lenta*, and up to the present regarded as yielding only methyl salicylate and glucose on hydrolysis (*cf.* Schneegans and Gerock, *Arch. Pharm.*, 1894, **82**, 437), is shown to be identical with the monotropitin isolated in 1923 by the author from *Monotropa hypopitys*, this giving methyl salicylate, glucose and xylose on hydrolysis. T. H. P.

Histological and Chemical Examination of the Seeds of *Ipomœa hederacea* and other species of *Ipomœa*. H. L. Kassner. (*Pharm. J.*, 1924, **112**, 328–331.)—An examination was made of the seeds of *Ipomoea hederacea*, Jacq., *I. pes-tigridis*, L.; *I. muricata*, Jacq., *I. coccinea*, L.; *I. pilosa*, Sw.; *I. eriocarpa*, R. Br., specially collected at Dehra Dun, India, and also of the commercial seeds of *I. muricata*, Jacq. and *I. hederacea*, Jacq., as sold for medicinal use in London. It was found that the oil content on the dried seeds varied from 14·94 per cent. in the case of *I. coccinea* to 9·23 per cent. with *I. eriocarpa*, and that in the collected and commercial samples of *I. muricata* and *hederacea* the variation was only from 13·08 to 12·21 per cent. The content of crude resin, however, was considerably higher in the case of the official *I. hederacea* (15·98 per cent. for the commercial and 15·59 per cent. for the collected seeds) than for *I. muricata*, which contained only 10·84 per cent. in the case of the commercial sample and 10·41 per cent. in the collected seeds. *I. pes-tigridis* contained the least proportion of crude resin (9·20 per cent.). The constants of the extracted resins and oils were as follows:

	Saponification value.	Acid value.	Refractive index. (n_D^{25})
I. hederacea, collected.			
Oil	191·0	3·8	1·4714
Resin	144·7	30·5	—
I. muricata, collected.			
Oil	185·4	3·5	1·4748
Resin	116·7	31·6	—
Commercial.			
Oil	184·5	—	1·4745
Resin	116·8	—	—
I. pes-tigridis, L.			
Oil	18?·8	3·3	1·4715
Resin	143·5	35·0	—
I. coccinea, L.			
Oil	193·4	3·7	1·4714
Resin	145·7	32·7	—
I. pilosa, Sw.			
Oil	196·7	4·1	1·4714
Resin	145·8	34·0	—
I. eriocarpa, R. Br.			
Oil	192·3	3·2	1·4728
Resin	144·6	25·0	—

The saponification values of the resins were determined by dissolving 0·5 grm. of the crude resin in 50 c.c. of cold neutralised 50 per cent. alcohol, diluting the solution with 750 c.c. of cold water (free from carbon dioxide), adding 0·5 c.c. of 1 per cent. phenolphthalein solution, and titrating the whole with 0·2 *N* alcoholic potassium hydroxide solution. The crude resins were found to give positive reactions in Kobert's tests for saponins, and the presence of saponin-like bodies in the resins is therefore inferred. D. G. H.

Analysis of Lysol. A. H. Dodd. (*J. Soc. Chem. Ind.*, 1924, **43**, 93–96 T.)—The author has examined the various methods adopted to effect the separation and measurement of the tar acids, including the separation of phenols by steam distillation, as recommended by the German Pharmacopœia and the method of Jordan and Southerden (*Pharm. J.*, 1921, 47a), and Chapin's method of extracting the tar acids with benzene, forming the phenates by adding 1 :2 sodium hydroxide solution to the benzol solution, evaporating the bulk of the benzene and decomposing the phenates with acid. Although the distillation method of separating tar from fatty acids, etc., is not generally recommended, such divergent results were obtained by other methods that the author carried out lengthy trials, and after applying corrections obtained results accurate to within 0·5 per cent. One hundred grms. (if possible) of lysol are measured into a separator, shaken with sufficient 2 per cent. sulphuric acid to destroy the emulsion and separate the fatty acids, 50 c.c. of ether added, the supernatant layer separated, and the dilute acids again washed with 20 c.c. of ether. The ethereal solution of fatty acids and phenols is dried over fused sodium sulphate (otherwise a sharp separation on distillation will not be obtained), filtered into a 250 c.c. distillation flask, and washed in with ether. The ether is distilled, and the fraction passing over between 100° and 160° C. tested for ether. The phenols are then slowly distilled at the rate of 1 to 2 drops per second, the flask and burner being completely enclosed in a metal cylinder, and the thermometer projecting from a sheet of asbestos used as a covering. The temperature gradually rises to a maximum at about 215° C., the rate of distillation falls and the temperature drops, phenols distilling slowly all the time. The flame is then gently raised till the temperature exceeds the former maximum by about 5°. After more drops have come over the temperature will again fall, and, on the third time of heating, 230° C. can be reached, and impure cresylic acid comes over. After this, an increase in the size of the flame will send the temperature rapidly up to 300° C. The necessary corrections were obtained by finding the percentage of cresylic acid distilling with mixtures of the usual quantities of oleic and cresylic acids (1) temperatures to 225° C. and percentages obtained on first heating; (2) to 220° C. after the temperature falls and the flame is raised; (3) on slowly distilling to 230° C. The results are given in a table. A comparison of the ordinary direct distillation method, Jordan–Southerden's, and Chapin's methods was made on 10 different brands of lysol, and the results (given in a table) show the need for an official method, since they differ from about 2 to 12 per cent. according to the method used. D. G. H.

Reactions of Dial-Ciba. Isnard. (*J. Pharm. Chim.*, 1924, **29**, 272–278.)—Dial-Ciba (diallylbarbituric acid) consists of small white anhydrous crystals melting at 170° to 171° C. It is very soluble in acetone, ethyl acetate and hot alcohol, and one part dissolves in 30 of cold 95 per cent. alcohol and in 20 of cold ether. Ten per cent. sodium hydroxide solution dissolves it readily; it is precipitated from the solution by acetic acid, but redissolves on addition of excess of acid. A 1 per cent. solution does not reduce Fehling solution; on melting the solid with potassium hydroxide ammonia gas is evolved; when heated for an hour with *N* alcoholic potassium hydroxide solution under a reflux condenser a solution is formed liberating gas on the addition of sodium hypobromite. On driving off the alcohol a precipitate is formed on the addition of hot water. Apparently, saponification begins and urea and malonic acid are formed. In common with other hypnotic derivatives of barbituric acid, the dixanthylated derivative (m. pt. 242–243° C.) is formed by condensation with xanthydrol in acetic acid. Large tabular crystals are formed on adding the necessary amount of hydrochloric acid to a solution of dial-ciba in 10 per cent. sodium hydroxide solution; one drop of a solution in dilute ammonium hydroxide solution mixed with one drop of ammoniacal silver nitrate solution results in the formation of prismatic crystals, isolated and grouped incrosses, squares and rosettes. Gardenal, the melting point of which is very close to that of dial-ciba, gives, under similar conditions, a granular precipitate composed of small isolated globular crystals, quite different from those from dial-ciba. Fuming nitric acid produces a violent reaction with dial-ciba, giving a clear yellow solution, but has no effect on gardenal. Dial-ciba, when treated with Laffon's reagent (ammonium sulphoselenite), gives a salmon-pink colour in the cold and a reddish-brown colour on warming; and with Mandelin's reagent (ammonium sulphovanadate) it gives a slight pink coloration in the cold and a brown colour on warming, whilst with gardenal no colour is produced in either case in the cold, and only a slight brown or yellow green coloration on heating. Since the sodium salt of dial-ciba is alkaline to methyl orange, litmus and rosolic acid, these substances cannot be used for its estimation. The samples of dial-ciba examined had an iodine value of about 235, corresponding to a product of a purity of 96·2 per cent. The iodine value is estimated by dissolving 0·2 grm. of the substance in 20 c.c. of ethyl ether, and continuıng the operation as in the case of fats. D. G. H.

Isolation and Constitution of the Active Constituent of Dalmatian Insect Powder. H. Staudinger and L. Ruzicka. (*Helv. Chim. Acta*, **1924**, **7**, 177–259.)—The substance pyrethron isolated as the active constituent of the flowers of *Chrysanthemum cinerariifolium* by Fujitani is not a pure substance, and the physiological properties ascribed to it are incorrect, on account of the impurities present. By extraction of the insect powder with petroleum spirit and shaking out the solution with aqueous methyl alcohol, carefully neutralising any acidity with potassium hydroxide, about 0·5 per cent. of an oily ester was obtained, which was separated into two parts by distillation *in vacuo*. The active part, which was strongly insecticidal, was a thick yellow oil having b.pt. 120–150° C. *in vacuo*. This fraction contains the pyrethrol of Fujitani, is readily saponified by alkali, and thereby rendered inactive. It is shown to contain two active constituents, namely, pyrethrin I and II, which are the methyl esters of chrysanthemum mono- and di-carboxylic acids, respectively, and are present to the extent of about 50 and 40 per cent. in the active oil, partly combined as semi-carbazones. These esters are exceedingly toxic to insects, mites being killed in about 20 minutes at a dilution in an inert powder of 1:10000. The synthesis, constitution, and reactions of the pyrethrins are described in detail. The proportion of pyrethins present in insect powder is about 0·2 to 0·3 per cent., which figure is confirmed by comparing the toxicity of the powder with that of an artificial mixture containing 2 grms. of pyrethrin per kilo. of inert powder. No insecticidal compound was found in the flowers of *Tanacetum vulgare*, *Matricaria chamomillae*, or *Achillea millefolium*. H. E. C.

Biochemical, Bacteriological, etc.

Precipitation of Lacto-Proteins by Copper Salts. A. J. J. Vandevelde. (*Bull. Soc. Chim. Belge*, 1923, **32**, 376–386.)—A survey of the literature relating to the composition of the precipitate formed on adding copper salts to protein solutions shows divergent results, the proportion of copper in the precipitate being given as from 0·7 to 20 per cent. The differences are mainly due to adsorption. According to Ritthausen lacto-protein gives a precipitate containing 16 to 17 per cent. of CuO, but, by using chloride, sulphate, acetate, and nitrate of copper as precipitant and varying the concentrations and the amount of water used for washing the precipitate, the copper percentage varies from 1·6 to 4·1. Examination of the solid and liquid phases shows that the equilibrium is influenced by the initial concentration; no definite compound is formed. When copper sulphate is the precipitant the cation is the active part, the anion being only adsorbed in traces by the precipitate. Harnack's results, which apparently showed that compounds were formed containing 1 or 2 atoms of copper to the molecule, according to the concentration, are found to be erroneous. H. E. C.

Tryptophan Content of Caseinogen. H. Onslow. (*Biochem. J.*, 1924, **18**, 63–84.)—None of the previous attempts to estimate tryptophan has been successful. In an earlier paper (*Biochem. J.*, 1921, **15**, 392) the author describes the compound precipitated by mercuric sulphate from a caseinogen solution which had been digested by enzymes. Since it was found that the total nitrogen of the precipitate was no criterion of the tryptophan content, owing to the other amino acids present, an attempt was made to obtain a value for the tryptophan from the difference between the amino nitrogen and the more resistant nitrogen of the pyrrole ring. The following equations were used:

$$\text{Total } N = \text{tryptophan } N + \text{histidine } N + \text{monamino } N + \text{peptide } N.$$

$$\text{Amino } N = \text{tryptophan } N/2 + \text{histidine } N/3 + \text{peptide amino } N + \text{monamino } N.$$

$$\text{Non-amino } N = \text{tryptophan } N/2 + \text{histidine } 2N/3 + \text{peptide non-amino } N.$$

$$\text{Or Tryptophan } N = 2\ (\text{non-amino } N\text{-(histidine } 2N/3 + \text{peptide non-amino } N)).$$

The non-amino nitrogen can be calculated from the difference between the total nitrogen (obtained by Kjeldahl's method) and the amino-nitrogen (obtained by Van Slyke's method); the histidine nitrogen by the methods of Koessler and Hanke (*J. Biol. Chem.*, 1919, **39**, 497); the peptide non-amino nitrogen from the difference between the amino nitrogen before and after complete hydrolysis; then the tryptophan content may be calculated from these. Difficulties encountered during the course of the work were overcome and 6 of the last 8 estimations out of 20 altogether were considered satisfactory. Four of these, carried out on one sample of caseinogen (English "casein") gave concordant values. The remaining two, carried out on another sample (Merck's "casein"), gave concordant, but rather higher, values. Full details of experimental procedure are given with a table of the results. The possible sources of error are discussed.

P. H. P.

Action of Rennet and of Heat on Milk. N. C. Wright. (*Biochem. J.*, 1924, **18**, 245–251.)—The previous work done on this subject is summarised and experimental details are given of the author's work, with tables of his results. The action of rennet and of heat on caseinogen solutions has been investigated by means of a study of the racemisation curves of the raw and treated protein. Caseinogen and casein have identical racemisation curves. This supports the view that rennet does not cause any proteolytic cleavage of the caseinogen molecule, but that coagulation is due to an alteration in the colloid state of the caseinogen, by which precipitation in the presence of bivalent metal ions is facilitated. Heat does not affect the constitution of the caseinogen molecule within the limits of the temperature studied (up to 120° C. for half an hour). Any difference in digestibility must be due to alteration in the physical (colloid) condition of the protein. The coloration of autoclaved milk is due to the caramelisation of the lactose, a reaction which appears to be catalysed by the presence of a colloid substrate of calcium caseinogenate. The colour of caseinogen precipitated from autoclaved milk is due to adsorption of this pigment and not to alteration in the caseinogen molecule itself. P. H. P.

Separation of a Fat Pigment from Fat. A. N. Currie. (*Biochem. J.*, 1924, **18**, 231–234.)—Several unsatisfactory methods of separation are discussed, but more efficient methods depend on hydrolytic treatment of the fat. The authors use calcium oxide and barium hydroxide as hydrolytic agents, and the calcium oxide gives the better results, since with barium hydroxide there is difficulty in separating the barium salts of the fatty acids present from the pigment. If the autoclave is used, at 12 atmospheres 1 per cent. by weight of lime is sufficient to effect complete hydrolysis. Full details of the method are given, and a scheme of the separation. Fat (from the abdominal wall of stall-fed cattle) was hydrolysed with lime, and the aqueous extract obtained then contained glycerol, pigment salt and small quantities of a fatty acid and the calcium salt of fatty acid. The fatty acid was removed by extraction with ether, leaving an aqueous layer containing pigment salt and glycerol. This was acidified with 20 per cent. hydrochloric acid and extracted with ether. The glycerol and calcium chloride were left in the new aqueous layer and the adipochrome (adipose tissue pigment) migrated immediately to the ether layer, showing that adipochrome freed from combination with calcium is soluble in ether. P. H. P.

The Antiscorbutic Fraction of Lemon Juice. S. S. Zilva. (*Biochem. J.*, 1924, **18**, 182–185.)—This work was carried out in continuation of the results already published by Zilva (*Biochem. J.*, 1923, **17**, 410). A great part of the solids of decitrated lemon juice consists of invert sugar. Details are given of a biochemical method devised for removing the sugar without appreciably altering the antiscorbutic activity of the solution, namely, by fermenting the sugar with yeast in an atmosphere of carbon dioxide. The preparations were tested for their antiscorbutic content on guinea-pigs and a table shows the results. It was further found that the antiscorbutic factor in the fermented decitrated lemon juice behaves in alkaline solution in the presence of air similarly to that in unfermented decitrated solutions. The alkali and air have an inactivating influence. The remaining active fraction, after removal of the sugar, contains less than half of the dry matter of the decitrated juice and is a complex mixture of substances, mostly of a nitrogenous character. Fermented decitrated juices give negative biuret, sulphur and tryptophan tests, but usually a faint murexide reaction masked by a brown coloration. Precipitates are obtained with mercuric sulphate, basic lead acetate and, occasionally, phosphotungstic acid; also Millon's reagent gives a precipitate which is soluble in excess of the reagent. The solution reduces ammoniacal silver nitrate and gives a Pauli reaction. No amide nitrogen can be detected. Very little extraneous matter is introduced by solution from the yeast, and most of the above reactions are also obtained in unfermented decitrated lemon juice. P. H. P.

The Vitamin *A* Potency of Hake Liver Oil. A. D. Holmes. (*Ind. Eng. Chem.*, 1924, **16,** 379–380.)—A daily dose of 0·8 mgrm. of crude hake liver oil is sufficient to meet the body requirements of growing albino rats fed on a diet lacking in vitamin *A*; hake liver oil is therefore equivalent to cod liver oil as regards vitamin *A* content. W. P. S.

Vitamin *A* Content of S. African Oils. E. M. Delf. (*Biochem. J.*, 1924, **18,** 93–100.)—The distribution of vitamin *A* has been studied in products belonging to the northern hemisphere, but very little is known of the vitamin content of the local produce of other countries. This work was carried out from a dietary point of view. Experiments on young rats are described and graphs of the results are given. The three vegetable oils, sesame, mafura and castor oils, were deficient in vitamin *A*. Two samples of seal oil had considerable growth-promoting properties. A difference in value was found in three different samples of crude whale oil. Sample No. 1, extracted at 100° C., was most active. The lower activity of the other two samples was correlated with their extraction at considerably higher temperatures. The sperm whale gave oils of differing activity with regard to vitamin *A*, that from the head being least and that from the blubber being most active in this respect. With more careful extraction, the blubber and meat oil would serve as a valuable source of this vitamin. The rat has little need of vitamin *C* in the diet, but a greater need of vitamin *A*. When the latter is limited the addition of vitamin *C* to the diet appears to have a really beneficial effect on growing rats. This suggests that the two vitamins are to some extent interchangeable in the diet of the rat. P. H. P.

Estimation of Sugar in the Blood. R. V. Stanford and A. H. M. Wheatley. (*Biochem. J.*, 1924, **18,** 22–28.)—The chief object of this note is to point out a source of error in this estimation as commonly practised. The method generally used is the modified one of Folin and Wu (*J. Biol. Chem.*, 1920, **41,** 367). The blue coloration obtained by the action of phosphomolybdic acid on cuprous oxide in acid solution gradually fades, and it is found that the colorimetric measurement must be made within an hour of the production of the blue colour, although short intervals make no perceptible difference. The reason why duplicate standards, made at the same time, often differ considerably in tint, however, is the temperature of the liquid when the blue colour is formed. The colour is darker the lower the temperature. Therefore the temperature at which the blue coloration is produced must be the same for the standard and for the unknown solution to be compared with it, and this is obtained by immersing the tubes in a cold bath. The authors give their own process and results obtained with the use of solutions described by Folin and Wu. A fallacy is pointed out in the results of Calvert (*Biochem. J.*, 1923, **17**, 117) due to his using an insufficient concentration of the alkaline copper tartrate. Therefore the reacting substances (especially the tartrate) must not fall below the concentrations given by Folin. The authors have carried out tests in which added dextrose was quantitatively recovered from a blood filtrate, and in which the content in reducing sugar of a blood filtrate was shown to remain sensibly unchanged after 2 days. The comparisons must be made in a dilution colorimeter.

P. H. P.

Detection of Incipient Putrefaction in Fish. J. Tillmans and R. Otto. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 25–37.)—The decomposition of fish differs from that of mammalian flesh; the criteria of soundness or otherwise therefore require modification. The commencement of putrefaction may be recognised by the estimation of ammonia and amino-acid nitrogen, and is indicated when these are more than 100 and 30 mgrms. per cent. respectively. The double titration method of Willstatter and Waldschmidt-Leitz (ANALYST, 1922, **47**, 308) also gives good results; when the difference between the acidity of a 10 per cent. extract in water and in 50 per cent. alcohol exceeds 3 c.c. of *N* alkali, decomposition has commenced. Another satisfactory test is oxygen absorption; 5 grms. of the minced fish are incubated in a closed bottle of water at 22° C. for 1 to 2 hours, the complete disappearance of dissolved oxygen indicating putrefaction. The reduction of methylene blue or of nitrates is a less delicate test, but their complete reduction in an hour or 2 to 4 hours, respectively, is a positive sign. Methods dependent on the detection or estimation of peptones, carbon dioxide, indol or soluble nitrogen, or on the iodine absorption yield indefinite or negative results.

H. E. C.

Bacterial Decay of Textile Fibres. A. C. Thaysen and H. J. Bunker. (*Biochem. J.*, 1924, **18**, 140–146.)—Results are given of problems put forward by Fleming and Thaysen (*Biochem. J.*, 1921, **15**, 407) when they described a swelling test for the quantitative determination of the destruction of cotton fibres caused by micro-organisms. With the use of this test it is shown that raw cottons of different origin vary in their resistance to this destruction. The resistance is probably due to difference in the physical or chemical nature of the respective fibres; sometimes the absence of food substances, such as phosphates, increases it. Since the rate of destruction of each type is practically constant, it has been possible, on several occasions, to identify raw cottons by means of this rate of destruction by micro-organisms. It is concluded that the resistance is adversely affected by climatic or soil conditions, since it disappears when resistant strains are cultivated in a country producing non-resistant types. Flax fibres appear to show differences in their behaviour to bacterial destruction similar to those exhibited by cotton. The rate of deterioration of jute and hemp fibres cannot be determined by the swelling test owing to the individual fibres being too interwoven. Little information is available as to the nitrogen content of raw cottons; some estimations by the authors are given. P. H. P.

Toxicological and Forensic

Poisoning by Barium Sulphate administered for X-Ray Examination. E. Dinslage and Fr. Bartschat. (*Zeitsch. Unters. Nahr. Genussm.*, **1924**, **47**, 7–9.)—The administration of 50 grms. of barium sulphate for an X-ray examination caused death in 12 hours; the barium sulphate was subsequently found to contain 90 per cent. of barium carbonate. For the estimation of the barium in the tissues the moist method of Fresenius was used, but as the oxidation of the organic sulphur leads to the formation of insoluble barium sulphate, the undissolved residue should be ignited and fused with fusion mixture, then extracted with water to remove sulphate, after which the residue soluble in hydrochloric acid is added to the main solution, and the barium identified and estimated in the usual way. In the case referred to the soluble barium was recovered mainly from the intestines, but small amounts were separated from the liver and kidneys. Tests on a number of samples of barium sulphate supplied for internal administration showed most of them to contain considerable quantities of soluble barium salts. The risk attending the use of barium sulphate for X-ray purposes is emphasised, and it is recommended that it be prohibited. H. E. C.

Failure of the Toxicological Tests for Phosphorus. Fr. Bartschat. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 10.)—Although yellow phosphorus in the free state or as phosphide can usually be detected many days after death, it sometimes happens that it is completely oxidised to phosphoric acid, in which case it is not revealed by the ordinary test. A case is reported in which 0·1 grm. of phosphorus was criminally administered in foods in small doses extending over two or three days; death ensued one week after the first dose, but phosphorus was not detectable in the post-mortem examination two days later, nor by chemical tests four days after the death. All the phosphorus had been oxidised in the tissues to phosphoric acid. H. E. C.

Forensic Importance of Blood-Groups. G. Roche Lynch and F. C. Martley. (*Lancet*, **1924**, **206**, 913.)—The relative numbers of blood-groups (first discovered in 1900) in the ordinary population are now fairly well known, as well as the broad differences found between Europeans and Asiatics. According to L. and H. Hirschfeld (*Lancet*, 1919, ii., 675) these were as follows:

	Cases.	Groups. Per Cent.			
		I.	II.	III.	IV.
Europeans.	Average of 4000	4·70	41·23	12·14	41·93
Asiatics.	Average of 2500	6·74	21·84	32·74	38·68

The most convincing theory advanced in explanation of the facts is that of Dungern and Hirschfeld, which postulates the presence of two agglutinogens and two agglutinins. The application of the test lies in two directions—(1) blood stains, (2) paternity cases. In the case of a man charged with murder it might be claimed by the prosecution that blood stains found on him were from the victim, and by the defence that they were from himself; the group of the stain would, in many instances, enable one or other of these suggestions to be negatived.

Tables by Dyke and others give the blood-groups of the offspring that might be produced by any combination of parents:

Parents.	Offspring may be—	Cannot be—
I. and I.	I., II., III., or IV.	——
I. and II.		
I. and III.		
I. and IV.		
II. and III.		
II. and II.	II. or IV.	I. or III.
II. and IV.		
III. and III.	III. or IV.	I. or II.
III. and IV.		
IV. and IV.	IV.	I., II. or III.

It would seem to be possible to demonstrate from the blood-groups that a given child could not have been produced by some particular parents—*e.g.* a Group II. child could not have sprung from two Group IV. parents. In applying the test to ascertain to which group a particular person's blood belongs a drop of II. serum and a drop of III. serum are placed at either end of a microscope slide, and a little of the blood under examination is taken up on a platinum needle and mixed with each. Agglutination can be readily seen with the naked eye in a few minutes; when it occurs in both drops the blood is of No. I. group; when it is in neither it is of IV. group; and when it is in only one it belongs to the group of the serum in which there is no agglutination. To ascertain the "titre" of a blood serum, drops of whole serum, half serum, quarter serum, one-eighth serum, and so on, up to about 1/250 serum, are treated with an equal volume of a 1 per cent. dilution of appropriate corpuscles, and the agglutinations noted under a low power of the microscope. They may extend nearly to the end of the series, or may only go down three or four places, the range varying greatly in different cases.

In applying the test to blood stains two methods may be used:—(1) An extract of the stain can be used to agglutinate known corpuscles, or (2) the agglutinating power of a known serum may be varied by treating it with unknown stain. The first is often difficult to demonstrate, but when a blood-stain on a garment is cut up into fine fragments and incubated in a sealed glass tube with a minimum quantity of water the resulting fluid, when filtered free from undissolved matter, generally agglutinates appropriate corpuscles when the preparation is kept overnight in a moist chamber. The second depends on the agglutinating power of a serum being finite. For instance, when a IV. serum which agglutinates II. and III. corpuscles is treated with II. corpuscles, it can still agglutinate III. corpuscles, as before, but its action on II. corpuscles is lessened, as shown by the agglutination "titre," or entirely abolished. In practice the blood stain is cut up and incubated as described above, except that in this case it is mixed with a known No. IV. serum instead of with water; after incubation the serum is drawn off and filtered, and its agglutinating power tested on known corpuscles. In the case of a IV. blood-stain no impairment of agglutinating power should be observed, but in the case of the other groups it can generally be detected. For instance, if the "titre" of the treated IV. serum was unchanged for III. serum, but very markedly diminished for II. corpuscles, it would be shown that the stain in question was No. II. blood. It should be noted that negative evidence alone may be fallacious, owing to the blood-stain having become inert from age or otherwise, or from its having been too small. In no case, however, has a definite positive result been found to lead to a wrong conclusion.

Organic Analysis.

Volumetric Estimation of Carbon. J. F. Durand. (*Comptes rend.*, 1924, **178**, 1193–1195.)—The following method for estimating carbon in organic compounds is applicable to a large number of substances, including probably all the hydrocarbons and ternary compounds containing carbon, hydrogen and oxygen. It lends itself to a method of micro-estimation to be described later. Use is made, as oxidising agent, of a sulphuric acid solution of permanganic anhydride, this being prepared by grinding in a mortar excess of potassium permanganate with about 20 c.c. of pure sulphuric acid of 66° Bé. The resultant green liquid, surmounted by oily droplets of the permanganic anhydride, is decanted and poured into one of the compartments of a calcimeter for measuring gases; about 2 per cent. of water is added to it. Into the other compartment of the vessel is introduced a solution of the substance to be examined (about 5 grms.) in 5 c.c. of sulphuric acid or carbon tetrachloride. The two liquids are then mixed, and the volume of carbon dioxide liberated is read over mercury at ordinary temperature. The liquid remains green if sufficient of the oxidising agent has been used. If the reaction is too violent, the oxidising liquid may be suitably diluted, since the anhydride must be prevented from liberating free oxygen and from assuming an appreciable vapour pressure owing to the thermal effect of the reaction. Of nitrogen compounds, phthalimide, azobenzene, dimethylaminoazobenzene, and picric acid yield virtually theoretical results, but urea, propionamide, and nitromethane yield no gas, and with some other compounds the carbon dioxide formed corresponds with only certain of the carbon groupings of the molecule. T. H. P.

Identity of Phocœnic and Valeric Acids. E. André. (*Comptes rend.*, 1924, **178**, 1188–1191.)—The supposed identity of Chevreul's phocœnic acid, prepared in 1817 from the oils of the dolphin and porpoise, with isovaleric (isopropylacetic) acid is confirmed. T. H. P.

Action of Bromine on Methyl Esters of Phenolsulphonic Acids. Estimation of Sulphur in Phenolsulphonic Esters and Salts. L. J. Simon and M. Frèrejacque. (*Comptes rend.*, 1924, **178**, 1282–1284.)—The authors have applied Desvergnes' method for analysing sulphonic acids (ANALYST, 1920, **45**, 386) to the methyl esters of various phenolsulphonic acids. The ester was first boiled with water until dissolved and hence hydrolysed, and the solution was then heated with a saturated solution of bromine in hydrochloric acid (20 c.c. for 2 to 3 grms. of the substance) until evolution of bromine ceased. The liquid was next diluted and filtered, and the sulphuric acid in the filtrate estimated. The method yields satisfactory results with the esters examined, and with the water-soluble additive products of the sulphonic esters with tertiary bases. The acyclic methanesulphonic and sulpho-acetic acids, or rather their barium salts, give no trace of sulphuric acid under the above conditions.
T. H. P.

Some Colour Reactions of Wool. Meunier and Rey. (*J. Soc. Leather Trades Chem.*, 1924, **8**, 149.)—To obtain the violet-red colour produced by the action of quinone on wool it is essential that moisture be present. If the wool has previously been exposed to the sun or ultra-violet light, the colour produced is dark brown, which may explain certain anomalies observed in dyeing fabrics which have been dried in full sunlight. Wools of acid reaction fix quinone more slowly, but, if previously exposed to ultra-violet light, are immediately coloured a bright yellow by quinone. Wools bleached by sulphurous acid are directly coloured bright yellow by quinone without exposure to sunlight, thus providing a ready method of testing whether a wool has been bleached with sulphurous acid. It is suggested that the sulphur in wool is partially oxidised by ultra-violet light. Wool bleached with sulphurous acid and then treated with a solution of sodium bicarbonate is again coloured violet-red. This colour reaction of quinone on wool is much quicker than in the case of silk, and thus affords a rapid test for the composition of a mixed fabric. Wool, whether bleached or not, if dipped in a neutral or alkaline solution of sodium nitrite, takes no colour at all, even if allowed to dry in the air; but, if dried at 100° C. for some hours, it is coloured an orange-yellow. If treated with sodium nitrite, exposed to ultra-violet light, and dried at the ordinary temperature, it is rapidly coloured orange-yellow, and this is more rapid if the sodium nitrite had been alkaline than if it had been neutral. (An identical reaction takes place with tyrosin.)

In the absence of nitrous acid Millon's reagent colours neither wool, silk, nor hair. In presence of small quantities of nitrous acid, wool, silk and hair are coloured red by this reagent, but the quantity of acid necessary is greater for hair than for wool, and greater for silk than for hair. If the acid is in great excess, no red colour appears, and there is immediate formation of a yellow colour. A very stable red colour can be obtained on wool or silk as follows:—*Silk*: From 0·1 to 0·2 grm. is dipped in a 7 per cent. solution of mercuric chloride. The solution is now warmed for 10 minutes and then five drops of a 25 per cent. solution of sodium nitrite are added. *Wool*: The same procedure is used as for silk, except that the solution is kept warm for 25 to 30 minutes before adding the nitrite.

To estimate the acidity of a wool 5 grms. are treated with 200 c.c. of 0·05 *N* sodium bicarbonate solution for 24 hours, with occasional shaking, and then rinsed four times with 50 c.c. of water. The mixed solution and washings are boiled with excess of 0·2 *N* sulphuric acid and back-titrated with 0·2 *N* sodium hydroxide solution. R. F. I.

Inorganic Analysis.

Rapid Electro-analytical Separation of Silver, Copper, and Bismuth by Graduated Potentials. A. Lassieur. (*Comptes rend.*, 1924, **178,** 847–849.)—The experiments here described were made with a fixed, cylindrical, platinum foil cathode, 43 mm. in diameter and 50 mm. in height, and a rotating anode, 30 mm. in diameter and 50 mm. high.

Silver and copper may be readily separated by electrolysing an almost boiling solution of the nitrates containing 1·3 c.c. of free nitric acid of 36° Bé. in a total volume of 85 c.c. and containing also a weight of mercury equal to about 10 per cent. of that of the silver. The latter is deposited first with a current of 2 ampères, the anode-cathode p.d. being 1·2 volt for the apparatus used; the time required is 20 minutes for each metal. The deposit of silver formed in the presence of the mercury is perfectly adherent and the cathode may be dried at 100° C. without loss of mercury. With silver and bismuth the operation is quite similar; during the deposition of the bismuth the auxiliary potential is maintained exactly at 240 millivolts, particularly at the end of the electrolysis; a good deposit of bismuth should be iron grey, matte and compact.

For the separation of bismuth and copper, an ammoniacal solution gives good results, the copper remaining dissolved and the bismuth being precipitated as hydroxide. The solution containing the two metals is treated with ammonia until it turns blue, an excess of 1 c.c. of ammonia (22° Bé.) and 10 c.c. of 95 per cent. alcohol being then added and the volume made up to 100 c.c. The solution is electrolysed in the cold with an auxiliary potential of 320 millivolts, which may be allowed to increase at the end of the electrolysis to 400 millivolts, the copper being weighed; for quantities of copper not exceeding 0·5 grm. this deposition occupies 20 minutes. The liquid is then neutralised by means of nitric acid of 36° Bé., 10 c.c. of which are then added in excess. The bismuth is then separated from the nearly boiling solution, the auxiliary potential being fixed at 240 millivolts.

T. H. P.

New Reagent for Carbon Monoxide. A. Damiens. (*Comptes rend.*, 1924, **178**, 849–852.)—When cuprous oxide (5 grms.) is slightly moistened with water and concentrated sulphuric acid (100 grms.) is gradually added, a reagent is obtained which readily absorbs carbon monoxide in considerable volumes, and may be used for the estimation of this gas in a gaseous mixture. Ethylenic and acetylenic hydrocarbons and oxygen are also absorbed to a slight extent and must, therefore, be removed before the carbon monoxide can be estimated in this way. The compound formed appears to have the formula, $Cu_2SO_4, 2CO$. When large proportions of carbon monoxide are absorbed by the reagent, the latter assumes an appreciable vapour pressure; hence it is necessary that treatment of the gaseous mixture with a second portion of the reagent should result in no further diminution in the volume. T. H. P.

Solution of Tin and Antimony Oxides. E. Stelling. (*Ind. Eng. Chem.*, 1924, **16**, 346.)—Tin and antimony oxides, even after they have been ignited, may be dissolved readily in hydrochloric acid if they are digested previously with water containing sulphur dioxide. The oxides are not reduced by the treatment, and the sulphur dioxide is not oxidised to sulphuric acid; a portion of the sulphur dioxide, however, combines with the oxides and is expelled by the hydrochloric acid. The following procedure is recommended for the treatment of an alloy containing tin or antimony, or both. One grm. of the sample is treated with 20 c.c. of nitric acid and 10 c.c. of water, the mixture evaporated to dryness, 10 c.c. of dilute nitric acid are added to the residue, and the tin and antimony oxides are collected on a filter and washed with dilute nitric acid. The oxides are then transferred to a beaker, digested for five minutes at 60° C. with 50 c.c. of concentrated sulphur dioxide solution, and the mixture then boiled, with the addition of 10 c.c. of concentrated hydrochloric acid, until all sulphur dioxide has been expelled. The tin and antimony oxides will now be in solution. W. P. S.

Estimation of Tungsten in Low-grade Ores. E. Jünger. (*Zeitsch. anal. Chem.*, 1923, **63**, 438–439.)—Two grms. of ore are fused with 4 parts of sodium peroxide in a nickel crucible. The fused mass is leached with 100 c.c. of water, the liquid filtered, and the precipitate well washed. The filtrate is heated, acidified with nitric acid, and made alkaline with ammonia; the precipitate is left to settle and filtered off. The hot filtrate, faintly acidified with nitric acid, is treated with excess of mercurous nitrate solution followed by ammonia until the precipitate gets black; the liquid, however, should remain faintly acid and contain mercurous nitrate. The precipitate is filtered off, washed with boiling water and dried, and the paper ashed separately. The precipitate is ignited to constant weight in a platinum crucible and fused with potassium bisulphate, and the cold mass is treated with hot water and a fragment of ammonium carbonate; if the resulting solution is not perfectly clear, the flocculent precipitate (silica or alumina) is filtered off, ignited and weighed, and the weight subtracted from the first, tungstic acid being obtained by difference. W. R. S.

Estimation of Iodine in Iodides. Weichherz and Klinger. (*Chem. Zeit.*, 1924, **52**, 269.)—This method can be used in presence of chlorides and bromides. It depends on the fact that iodides in acid solution are oxidised by potassium permanganate, the excess of which is then destroyed by means of oxalic acid, and the liberated iodine is titrated with sodium thiosulphate solution. In the reaction between the hydriodic acid and the permanganate hydrated oxide of manganese separates, in spite of the acid solution, but the presence of manganese sulphate facilitates its solution, accelerates the reduction of the permanganate and prevents small amounts of chlorine or bromine from interfering. If the amount of iodine is small and that of the chlorine or bromine is large, these, even though liberated, can be eliminated by adding formic acid, which reduces chlorine and bromine but not iodine. R. F. I.

Physical Methods, Apparatus, etc.

Analysis of Binary Mixtures. Volumetric Turbidity Method. C. D. Bogin. (*Ind. Eng. Chem.*, 1924, **16**, 380–386.)—Binary mixtures of two liquids, one of which has a much greater solubility in water than the other, may be analysed by titrating with water until a turbidity appears. The volume of water that can be added to a unit volume of a binary mixture of any certain composition before turbidity appears is definite and invariable, independent of the time of contact, amount of shaking or rate of addition, and only slightly dependent on the temperature. Applications of the method to mixtures of ethyl and butyl alcohols, propyl and butyl alcohols, ethyl alcohol and petroleum, benzene and ethyl alcohol, etc., are described and the curves for the various mixtures are given. For the analysis of a mixture of ethyl and butyl alcohols, 20 c.c. of the sample, dried previously over potassium carbonate, are placed in a flask and water is added from a burette until a turbidity appears; the end-point is quite sharp (within 0·05 c.c.), and reference to a curve gives the composition of the mixture. The method may be applied to ternary mixtures if an independent method is available for estimating the proportion of the third constituent, and mixtures of organic liquids and water, salt solutions, and pure organic liquids may be used as titrating media in the same way. Mixtures of two liquids, both of which are miscible with water, may be analysed by adding a definite volume of a third insoluble liquid and then titrating with water; mixtures of solids may be analysed by dissolving them in a liquid and then titrating with another liquid. The percentage of water in organic liquids (*e.g.* acetone, butyl alcohol) may be estimated by titration with benzene, and moisture in solids may be estimated by extracting the sample with an organic liquid and then titrating the latter with benzene. W. P. S.

Estimation of Silicon in Iron-Silicon Alloys by their Physical Properties. T. D. Yensen. (*Ind. Eng. Chem.*, 1924, **16**, 366–367.)—The electrical resistance and hardness of iron-silicon alloys are functions of their silicon content. The relationship between silicon and electrical resistance is shown by a curve consisting of two straight lines with a bend at Si 0·35 per cent., and may be expressed by the equations:

$$\text{For Si} \leqq 0{\cdot}35 \qquad p = 9{\cdot}6 + 18{\cdot}4 \times \text{Si}.$$

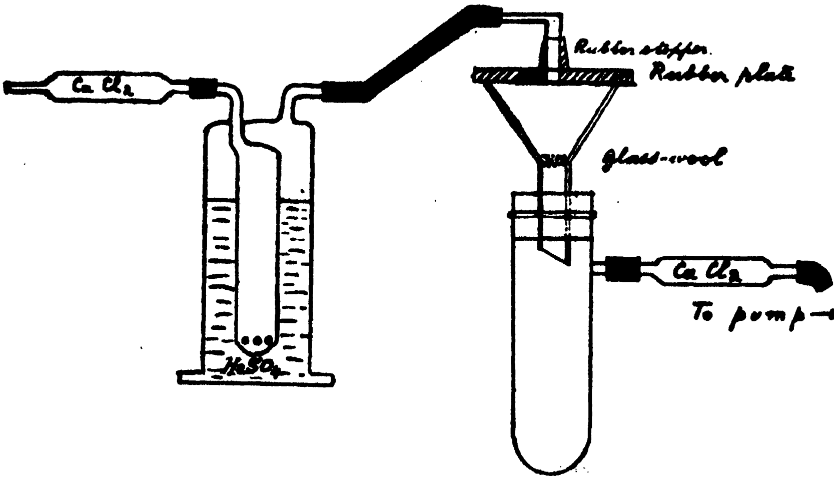
$$\text{For } 0{\cdot}35 \leqq \text{Si} \leqq 6{\cdot}5 \qquad p = 16{\cdot}05 + 11{\cdot}1\,(\text{Si} - 0{\cdot}35).$$

The curve showing the relation between silicon and hardness may be expressed by the equation:

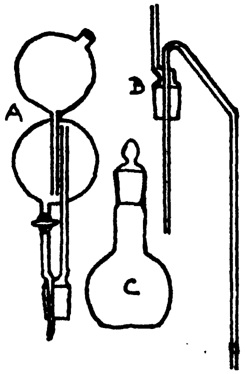
$$\text{Si} = 0{\cdot}092 \times (N - 86)^{0{\cdot}777}$$

where N = the Brinnell hardness number (or P/A), P = load, in kilos., on a 10 mm. diam. steel ball, and A = the area, in sq. mm., of the impression made by the 10 mm. ball on the sample. Either method will give the silicon content within 0·1 per cent. W. P. S.

Apparatus for Filtering Hygroscopic Substances. Ban. (*Chem. Zeit.*, 1924, **52**, 271.)—The rubber plate with stopper shown in the diagram is pressed by means of a stand and clamp on to the rim of the funnel, the ring of contact being smeared with desiccator-grease. In the case of oxidisable substances an inert gas, or air freed from oxygen by means of alkaline pyrogallate solution, can be drawn through the apparatus. R. F. I.



Apparatus for the Analysis of Oleum and other Fuming Liquids. E. Bosshard. (*Helv. Chim. Acta*, 1924, 7, 330–332.)—The stoppered flask C, made of resistant glass and of about 150 c.c. capacity, is weighed; then the stopper replaced by the tube B, the end of which dips into the liquid to be analysed and of which about 5 grms. are introduced by suction. B is removed without wetting the neck of the flask, the stopper replaced and the weight noted. The bulb head A is now attached; the upper bulb contains glass beads, and the lower one about 100 c.c. of water. The flask C is cooled in ice, and the apparatus inclined so that drops of water introduced from A run down the side of C; water is then very slowly added from A to dilute the oleum; any sulphur trioxide evolved is trapped and absorbed in the head A. When sufficiently diluted, the liquid is made up to a definite volume and an aliquot part titrated in the usual way. When chlorosulphuric acid is being analysed it is a good plan to put about 5 times its weight of ignited sand (previously washed with hydrochloric acid) into C before weighing the acid. The apparatus is recommended for use with a number of other fuming acids and phosphorus compounds. H. E. C.



Reviews.

General Chemistry. By H. G. Deming. Pp. 605. New York: John Wiley & Sons; London: Chapman & Hall, Ltd. 1923. Price 17s. 6d. net.

When a further addition is made to the already too large number of text books of general chemistry one may reasonably search for the distinctive feature or merit which alone can warrant a new publication. Prof. Deming sets out to treat of separate principles in each chapter and to illustrate them from industrial applications, thus avoiding the cataloguing of the ever-increasing number of compounds, many of which are of purely academic interest. As the volume touches on organic, physical and biological chemistry, as well as its principal subject of inorganic chemistry, it will be realised that it has been necessary to omit the detailed description of a number of substances usually included in a book of this type.

The result has been to produce a very readable treatise in which the general principles and applications of chemical science are clearly presented. The more important recent developments, such as X-ray analysis and the Lewis-Langmuir theory of atomic structure, are described in an explicit, though elementary, manner, and in such a way as to stimulate clear thinking and further investigation.

A book with such a wide scope is sure to contain blemishes. The description of some of the industrial processes is meagre and too scrappy to be of much value;

for instance, the hydrogenation of oils is dismissed in about five lines, and the preparation of hydrogen is discussed in the ordinary text-book manner, after which it is hardly fair to ask the student at the end of the chapter (p. 320) "if you were to establish a small plant for the hardening of vegetable oils what sources of hydrogen would you consider?"

There is an American style about the book, but the general presentation is one which will be acceptable to English readers, though it may be regretted that English spelling is not adhered to in words such as "sulfur" and "tires." The printing, paper and binding are good, but some of the illustrations are not all that they might be.

H. E. Cox.

CANNED FOODS IN RELATION TO HEALTH (MILROY LECTURES, 1923). By WILLIAM G. SAVAGE, B.Sc., M.D. (Lond.), D.P.H. Cambridge Public Health Series. Pp. vii. +146. Cambridge: University Press. 1923. 8s. 6d. net.

Those who are acquainted with Dr. Savage's work on the bacteriology of canned foods will welcome the appearance of this book, which is based on the Milroy Lectures for 1923 delivered by the author before the Royal College of Physicians. Four or five years ago Dr. Savage set himself the task of undertaking a thorough investigation into the bacteriology of canned foods, a subject about which there had been much misconception and difference of opinion. As recently as the year 1920 we find definite statements by various authors that the keeping qualities of canned foods, with the exception of sweetened condensed milk, depend on their absolute sterility. As a consequence of Savage's work this view is no longer tenable. Of the foods examined by him a large proportion were by no means sterile. Over 60 per cent. of canned meat samples and of marine products contained living spores, and no class of product was absolutely free from organisms.

The author points out that the keeping qualities of canned foods depend, not on sterility or non-sterility, but on whether the conditions within the tin are such as to prevent the multiplication of any organisms which may be present. What these conditions are, and how they affect the growth respectively of aerobes and anaerobes is fully discussed, and Dr. Savage's arguments, supported as they are by a wealth of experimental data, seem to be conclusive.

Food poisoning due to specific bacteria and their toxins naturally receives extended treatment, and it is worthy of note that only a month before the outbreak of botulism at Loch Maree in 1922, Dr. Savage had referred at the Plymouth Congress to the possibility of this deadly form of food poisoning being introduced into this country. Savage's work enables us to draw the broad conclusion that bacterial food poisoning from canned foods is due almost entirely to certain specific bacteria which have gained access to the food prior to canning, owing to lack of care and cleanliness in the preparation of the food, and that the occurrence of these specific bacteria is comparatively rare. The ordinary decomposition of food, *e.g.* by organisms of the *B. proteus* group, has never been shown to be associated with food poisoning, nor is there any evidence that the so-called "ptomaines" are injurious to health. At the same time the author makes it clear that tins which are "blown," or which show obvious decomposition of their contents, are evidence of faulty methods of preparation or storage, and are on that account to be rejected as being the more likely to have been exposed to infection by specific bacteria.

While the bacteriologist can hardly fail to agree with the author's conclusions, the chemist will feel that the factors influencing the action of foodstuffs upon tin, and the possibilities of tin-poisoning occurring through the consumption of canned foods, have been dismissed in somewhat too arbitrary a manner. A number of recorded cases of alleged tin-poisoning were discussed by Buchanan and Schryver in a Report to the Local Government Board in 1908, but Savage is of opinion that the evidence pointing to tin as being the active agent is by no means conclusive in many of these cases. This may be so, and, further, it is true that neither Lehmann nor Schryver could find any evidence of chronic tin-poisoning resulting from the ingestion of canned foods over a considerable period. We are familiar with negative evidence of this character, not only in the matter of tin, but in the case of almost every impurity or chemical preservative commonly found in foodstuffs. Injury to health does not necessarily begin only with definite symptoms of poisoning, and it is clearly in the interest of the consumer that the amount of tin in canned foods be kept down to the lowest possible limit. Much yet remains to be done on the purely chemical side in investigating the mechanism of the action of different foodstuffs on tin-plate.

The book concludes with two appendices giving an account of some of the principles involved in the processing of canned foods and of the methods of examination in the laboratory. There is a full bibliography and a short index. Dr. Savage's work can be thoroughly recommended as an admirably concise and lucid exposition of modern views on the subject of canned foods.

G. W. MONIER-WILLIAMS.

OILS, FATS, WAXES AND RESINS. By E. R. BOLTON and R. G. PELLY. Pp. 275. (The "Resources of the Empire" Series.) London: Ernest Benn, Ltd. 1924. Price 21s. net.

This is one of a series of twelves volumes which have been prepared by the Federation of British Industries for the purpose of bringing before business men and others the Empire's resources. As is the case with each book in the series, it opens with a Foreword by H.R.H. the Prince of Wales, and a general Introduction by the Right Hon. Sir E. Geddes. In addition, there is an introductory review by Mr. J. H. Batty. The introductory matter, with the Author's Preface and Table of Contents, occupies 20 pages, and contains much of interest.

This book is partly statistical in nature, and probably for the first time authentic and official statistics on the subjects dealt with in this volume are available in a well-classified form under one cover. It must not be supposed, however, that, as the volume is largely statistical in form, it lacks interest or information of any other kind. On the contrary, it is replete with material concerning methods of collection, descriptions of varieties of commercial products, outlines of manufacturing processes, methods of valuation and chemical data that make the volume of all-round value to the business man with Empire-wide interests, as well as to the chemist, the student and the technical expert.

The book comprises, as its title indicates, commercial, technical and statistical information on oils, fats, waxes and resins; it defines and classifies these terms, discusses the sources of supply, and describes the commercial methods of manufacture of oils, fats and oil-cakes, etc. The refining of oils, production of fatty acids by splitting, and hydrogenation are described lucidly and concisely. Analytical data are supplied, the composition of the various products is given, and the methods of commercial valuation briefly set out. All the important varieties of these substances are adequately dealt with, for the most part in consecutive chapters, the whole constituting Part I. of the book, comprising 135 pages.

Part II. (100 pages) is concerned with the Production and Trade of the British Empire in these commercial products. Part III. deals with the development of Imperial resources of oilseeds and oils, and contains some valuable suggestions as to the way in which the less-known products may be exploited in the future. The difficulties which must be overcome before trade in certain directions can increase are duly emphasised. In a short special section Mr. H. Moore reviews the mineral lubricating oils in a manner similar to that done for the fatty oils in Part I. Part IV. contains an important list of officials and official institutions from which information is obtainable, and the work concludes with a bibliography containing references to some of the more useful publications on the various products described, and with an index of subjects and of botanical and zoological names.

The reviewer is impressed with the very considerable scope of the statistical information, and with its great value. This information is not only noteworthy for what it comprises, but also because it draws attention to respects in which our knowledge is scanty or non-existent, and so suggests the desirability of getting further facts relating to many important products. In this way the volume has done real service to the Empire, and the Federation of British Industries is to be congratulated on its scheme of inaugurating this well-conceived series. No such complete statistics of the kind have hitherto been available, and although the Bulletins of the Imperial Institute have for years given information of this character (and the authors acknowledge their debt to these bulletins), there is no other publication which combines the subject-matter in a digested form, suggestive and interesting, and provocative of ideas for the extension of the Empire's trade. The work will be invaluable to the business man whose interests lie within the subjects dealt with here; the technical chemist is provided with a mass of material which he must long have desired to have in a handy form. The authors also are to be congratulated on the way in which they have presented this material. The publishers have done their part to make the book presentable in form, while the printing is excellent. The proofs have evidently been carefully read, and very few errors meet the eye. The volume makes a timely appearance during the early days of the British Empire Exhibition. ARNOLD R. TANKARD.

FATS: NATURAL AND SYNTHETIC. By W. W. MYDDLETON and T. HEDLEY BARRY. Pp. xi. +182. London: E. Benn, Ltd. 1924. Price 25s. net.

The authors of this book have made a useful addition to the literature of chemical industry by gathering, into the compass of one volume, much information, hitherto only available in scattered papers, on the preparation and commercial uses of the hydrogenated oils. The descriptions of large scale processes are clearly written, and the diagrams illustrating the text are good. The handling of the theoretical side of the subject is less satisfactory. The authors do not seem to have clearly in view whether they are writing for the manufacturer seeking a knowledge of chemistry, or for the chemist wishing to acquire a knowledge of manufacture. This results in an unfortunate confusion of style; for instance, while "saturated" and "unsaturated" acids are referred to in inverted commas on p. 7 and defined on p. 15 as compounds which take up bromine, in the former case with, and in the latter case without, displacement of hydrogen, yet the theory of the tetravalent carbon atom and of the single, double, and treble bond, is taken for granted throughout the book. Both the deductive method and the empirical method of teaching have their uses, but a mixture of the two is confusing.

A statement that cannot be allowed to pass uncriticised occurs in the preface, namely, that "the methods of oil analysis are not of the same order of accuracy as the standard methods of inorganic chemistry." This is more than misleading. The methods of oil analysis have perforce to be of a much higher order of accuracy than those used in ordinary inorganic analysis, since, owing to the much greater mass of the fatty acid molecule, the interpretation of the analytical results is so much less sharply defined.

In the later and less theoretical chapters, Chapter V. and onwards, the presentation of the matter greatly improves, and the authors are to be particularly complimented on Chapters VI., VIII. and XI. The setting out of the physical properties of fats in the form of graphs is a valuable innovation. The curves given in figure 26, however, appear to be unsatisfactory, since the iodine value for arachis oil, calculated from the percentage composition, is in two cases some twenty units higher than the value given at the head of the column on p. 107.

A few statements in the text seem to require qualification; *e.g.* on p. 32, cottonseed meal is referred to as used for human consumption; on p. 39, the fish liver oils are referred to as "valued almost entirely for medicinal properties"—dietetic would have been a better word than medicinal, and it has been overlooked that several thousand tons yearly are absorbed by the tanning industry. In the account of the preparation of edible oil from seeds, it should have been mentioned that the neutralised oil is deodorised by means of superheated steam.

In dealing with the commercial uses of the oils and fats the author discusses the function of these as human food. A brief account is given of the vitamin theory which, though correct enough in what is present, is vitiated by an absence of allusion to the subject of rickets. The rickets problem is admittedly one of the most pressing social problems of the day in this country, and is closely connected with an inadequate fatty diet. The conclusion that it does not "seem necessary to insist that they (the vitamins) should be present in it (a synthetic fatty food) at all, since a sufficiency apparently can be obtained from the other substances in the diet of the average person" is a piece of false argument, since it is amongst that very class of the population which suffers from a generally inadequate diet that there is also the tendency to substitute cheap margarines for butter. It is of the greatest importance therefore that these should be made as complete in dietetic value as possible.

In general, "Fats, Natural and Synthetic" may be commended for containing "the truth and nothing but the truth," but, unfortunately, not for containing the whole truth. As a work of reference the information which it gives is generally sound, but there are blanks the existence of which is not even indicated. The references given to original papers, while all useful, are too haphazard to be regarded as forming a bibliography.

The book, unfortunately, is not free from those defects of literary style which are so constantly attributed to scientific authors; for instance, on p. 10 "unique" is used where "single" is meant (it would have been exciting to meet a "unique" hydrogen atom!); on p. 31 "above" is used as a noun; on p. 103 "smallest" is assumed to be synonymous with "least possible"; and on p. 87 hydrogen is added "to the double bond." There are also difficulties with that subtle mood, the gerundive, and on p. 30, paragraph 6, a full stop appears somewhat surprisingly in the middle of a sentence. The paragraphing also needs revising, being without any obvious system. On p. 140 every sentence except one has a paragraph to itself.

Two other defects are the mixture of type founts used for chapter sub-headings, and confusion in the use of the sign " = ". This is used both for "equals" and for a double bond. At first sight it is not always clear which is meant; for instance, on pp. 10 and 11, "=C" is in the middle of an equation, and on p. 14, of a molecular formula. The consistent use of spacing round the "equals" sign, such as indeed is found in three out of the four equations on p. 11, would make the meaning clear at the first glance. All these minor defects could have been removed by a more careful attention to detail in the proof stage, and it is to be hoped that they will have disappeared in the second edition that is certain to be demanded.

The publishers, Messrs. Ernest Benn, Ltd., are to be congratulated on the general appearance of the volume. DOROTHY JORDAN LLOYD.

THE CHEMISTS' YEAR BOOK, 1924. Edited by F. W. ATACK, M.Sc., D.Sc., B.Sc., F.I.C. Ninth Edition. Pp. 1147 and Index. Manchester: Sherratt & Hughes. 1924. Price 21s.

Although the present edition of this work is larger by 40 pages than that of 1923, it is issued in the form of one volume, and in that respect is decidedly more convenient for frequent reference than its two-volume predecessors.

The abundant tabular matter has been supplemented with some new data, chiefly from American sources. One new table, giving the years and corresponding volume numbers of the more important scientific periodicals, should appreciably facilitate the often wearisome task of tracing references.

Other new features of the book include a section on "Essential Oils" by T. H. Durrans and one on "Spirits" by F. Robinson. The former is clearly written, and as full as could be expected in a work of this kind, and includes a short bibliography of the subject. The section on "Spirits" too, is well written, though it does not give sufficient information as to where fuller details can be found; there is a slip on p. 905, line 1, where "ammonium acetate" should read "aniline acetate." The section on "Water Analysis" has been re-written by S. E. Melling; it is considerably longer than in earlier editions, and is fairly adequate for laboratory reference. This section also contains a short bibliography.

An unfortunate error on p. 628, where mannitol and dulcitol are referred to as "sugars," might leave an inexperienced reader in doubt as to whether the alcohols named or the corresponding sugars should be used. The section on "Agricultural Chemistry" has been revised and slightly enlarged. It could hardly be further improved within the limits of space assigned to it, and is indeed a model of what special articles in works of this kind should be. Brief accounts of American methods of analysis constitute a new feature of this and several other sections. The section on "Dairy Products" also deserves high commendation.

In reviews of previous editions (ANALYST, 1921, **46**, 431; 1923, **48**, 354) it was pointed out that some of the sections dealing with special branches of analysis were too short to be of value in themselves, and that they gave few or no references to authorities. It is satisfactory to note that these defects have been remedied to a large extent, and that, although some sections are still open to complaint on these grounds, the book as a whole should prove a most useful laboratory aid.

LEWIS EYNON.

PHOTOGRAPHY AS A SCIENTIFIC IMPLEMENT. By Various Contributors. First Edition. Applied Physics Series. Pp. viii.+549. London: Blackie & Co. 1923. Price 30s.

This treatise, as stated in the preface, is an endeavour to include in one volume the "whole of the results achieved" in the development of methods of technique in various branches of science, and thus to make available to scientific workers methods with which they may be more or less unfamiliar, but which may be of service in their own particular branch. That this aim has not been attained will be obvious from the size of the volume, but the attempt has led to the production of a textbook which will prove not only of great value to scientists, but also of interest to the general reader. The title of the volume is perhaps not sufficiently comprehensive, since much is included that cannot be classed under the above heading, especially in optics and photographic research.

Chapter I., by Mr. C. R. Gibson, consists of a brief history of photography down to about the year 1850, with, in addition, short paragraphs on the wet collodion process and the gelatine dry plate. No reference is made to the evolution of the isochromatic or panchromatic plate, since this is dealt with elsewhere in the volume. Chapter II., by Mr. S. E. Sheppard, is devoted to the elementary optics of photography, and, although more or less a bald statement of facts, and therefore tedious reading, is none the less accurate and reliable. In Chapter III. the more advanced optics of the photographic lens is dealt with by Professor A. E. Conrady from the point of view of the practical photographer; it treats of the formation of images and the various aberrations and other factors limiting the formation of the ideal representation of an object photographed, in addition to experimental methods for determining the constants of any lens system. Chapter IV. provides the theory of photographic processes and methods and is also written by Mr. S. E. Sheppard. In this chapter the production and manipulation of photographic plates and papers are treated in a thorough manner including much recent work.

The preceding four chapters may be said to constitute Section I. of the volume, for the remainder of the book is devoted to the application of photography to various branches of science. In Chapter V., by Mr. C. R. Davidson, an admirable account is given of the use of photography in astronomical work, including descriptions of the apparatus used and the methods employed for the production of spectra and determination of luminosity of the heavenly bodies. Chapter VI. is contributed by Mr. H. Moss, and illustrates in a striking manner the great utility of photography in the study of physics. Both the methods adopted and the results achieved in Worthington's work on splashes, Boys' researches on bullets in flight, and many other branches of physical investigation are described and illustrated. Chapters VII. and VIII., the former by Mr. J. H. G. Monypenny, and the latter by Dr. G. H. Rodman, may be mentioned together, since both consist principally of admirable descriptions of practical manipulation in photomicrography, the first in its application to metallurgy and the second in its more general bearings. "Photographic Surveying," by Col. H. S. L. Winterbotham, is the subject of Chapter IX., and the methods adopted in topographical work, both from the ground and from aeroplanes, are described in a lucid and agreeable manner.

An interesting account of the development of the aeroplane camera is given in Chapter X. on "Aeronautical Photography," by Mr. F. C. V. Laws, in which the essentials required to achieve the greatest efficiency, together with methods of manipulation adopted for the production of single and stereoscopic photographs, are recorded.

Mr. W. L. F. Wastell, in Chapter XI., deals with the development of photography in colours and the various methods adopted at the present time for the production of photographs in the natural tints of the subject, a few pages at the end of the chapter being devoted to brief descriptions of the methods employed in colour kinematography. The extensive use made of photography in the production of illustrations of all kinds is made apparent by perusal of Chapter XII. by Mr. W. B. Hislop, in which the principles and technique of the different branches of relief, surface, intaglio and three-colour printing are described. This chapter is unique in that it is provided with an appendix giving formulæ and notes on the various solutions employed for development, intensifying, sensitising, etc.

Chapter XIII., entitled "The Technics of Kinematography," by A. S. Newman, is an able exposition of the development and present-day practice in the highly popular field of "the pictures." This subject is an excellent example of a lengthy and intricate process in which careful attention to detail throughout is essential to success.

The volume is brought to a close by Chapter XIV., "The Camera as Witness and Detective," by Mr. W. M. Webb, which, on the whole, is somewhat disappointing. Considering the enormous amount of material available, and the extensive variety of subjects included under this head, it is curious that much of the chapter deals only with the detection of erased writing on foreign passports. Although the matter included is of much interest, it is to be regretted that no examples are given of the use of photography in the study of watermarks, finger prints, forged paintings, the identification of ink and pencil markings, the deciphering of charred documents, and other applications frequently employed at the present time.

The index is perhaps the least satisfactory portion of the volume. The small "nonpareil" type employed is not conducive to a rapid search, this being in striking contrast to the subject matter which is printed in the much more legible "small pica." Further, several errors in the page references may be found, thus: "Astronomical photography" is not to be met with on page 398 as given, nor is "Autograph, stereo-" on pages 200 and 209–60, whilst, in addition, various subjects found in the text are not mentioned in the index, among these being "Backing of plates," "Compensation eyepieces," "Astrophysics," "Portrait lenses," and others. For these and other reasons it is to be hoped that the index will undergo a careful revision before the second edition is produced, when the opportunity might also be taken to introduce more complete cross-indexing than is at present provided in this volume.

In addition to numerous figures and diagrams interspersed throughout the text the volume contains over forty plates, many of which are excellent examples of the results obtained in various branches of photography. Unfortunately no index to these is provided, and, since both text illustrations and plates are arranged somewhat erratically, the omission is particularly noticeable.

One admirable feature of this production is the series of bibliographical references appended to each chapter, or in some cases to each section—an excellent example which might well be copied in other scientific manuals.

A perusal of the text shows that very few serious errors are present, but two have been noted. On page 203 the curves illustrating Purkinje's Phenomenon are wrongly lettered, thus suggesting that the converse of this is true, and, on pages 140 and 167, various expressions intended to represent chemical equations are decidedly unbalanced. Misprints are also few in number.

The information, both theoretical and practical, throughout the volume is of a high degree of accuracy, but the reviewer is unable to agree with the statement on page 322 referring to the photomicrography of opaque objects that "the photograph is better taken without such procedure" (bilateral illumination of the object), for undoubtedly the best results are achieved by a secondary diffused illumination on the side remote from the principal illuminant. The method given for the detection of changes in a complicated group of objects is not so recent as is indicated on page 312, for it was first described by Hodgson and Wilsey in 1917. Other and simpler methods for detecting such changes, which are not mentioned, are the stereoscopic observation of the two negatives and application of the method for the comparison of stellar photographs described on page 219.

The authors and publishers are to be congratulated upon the results of their efforts in breaking new ground and compiling a manual which is lucid, accurately expressed and not overburdened with the higher mathematics. It is to be hoped that we may look forward to a similar production dealing with the applications of photography in other branches of science not included in the present volume.

T. J. WARD.

The Institute of Chemistry of Great Britain and Ireland.

PASS LIST.

APRIL EXAMINATIONS, 1924.

The following candidates have passed the Examination for the Associateship:—*In General Chemistry*: R. P. Balfour, C. W. Gibby, F. J. Green, G. W. Harrison, A. G. Laing, G. L. McCarter, L. B. Milne, C. F. Myers, J. L. Philp, and W. E. Wright.

Under Regulations prior to March, 1920—*In the Chemistry, including Microscopy, of Food and Drugs and of Water*:—C. W. Maplethorpe.

The following candidates have passed the Examination for the Fellowship:—*In Branch D, Agricultural Chemistry*: B. Viswanath.

In Branch E, The Chemistry, including Microscopy, of Food and Drugs, and of Water:—G. M. Norman and R. G. Thin.

In Branch H, General and Analytical Chemistry:—C. Hollingsworth.

Publications Received.

THORPE'S DICTIONARY OF APPLIED CHEMISTRY. Vol. V. (Oxygen to Rye). Pp. 722. London: Longmans, Green & Co. Price 60s. net.

CLOUDS AND SMOKES. By W. E. GIBBS. London: J. & A. Churchill. 1924. Price 10s. 6d. net.

TECHNICAL METHODS OF CHEMICAL ANALYSIS. 2nd Edition. Vol. I. Edited by G. Lunge and C. A. Keane. London: Gurney & Jackson. 1924. Price 63s.

JULY, 1924. Vol. XLIX., No. 580.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The Effect of Giving Certain Oils in the Daily Diet of Cows on the Composition of Butter Fat.

By H. J. CHANNON, B.A., B.Sc. (Beit Memorial Research Fellow),
J. C. DRUMMOND, D.Sc., F.I.C. and J. GOLDING, D.S.O., F.I.C.

(*Read at the Meeting, March* 5, 1924.)

The work described in this paper is the result of the second part of an experiment planned with two objects in view:

(*a*) To obtain further and more exact information on the effect of cod-liver oil feeding of cows on the vitamin *A* content of the milk, than had been obtained in a similar experiment reported by Drummond, Coward, Golding, Mackintosh and Zilva (1923).

(*b*) To attempt to make a quantitative study of the composition of butter fat and the alteration in its composition induced by the inclusion of various oils in the daily diet of the cows.

The first part of the experiment on the vitamin content of butter is being reported in the Journal of Agricultural Science, 1924, but, owing to the fact that the conditions obtaining in the experiment were governed by the two objects in view, it will be necessary to make some reference to it in this paper. The effect of the administration of oil-cakes, oils and various special foods has been much studied, the method usually adopted being to follow the alterations in the composition of the butter fat by a determination of the usual analytical values of samples of butter fat from the cows under experiment. Further, iodised fats, or oils giving specific colour reactions, have been given in the diets of cows, and tests for iodine, or the colour reactions concerned, carried out on the butter-fat. This type of experiment is open to the objection that, even if iodine were found present in the butter fat, or the colour reaction found positive, it is still uncertain whether the oil itself, or the substance giving the reaction only, has passed into the butter fat; in the case of iodised fats, the presence of iodine in the butter fat does not necessarily mean that the iodine is still attached to the fat or other substance which was given in the food. Although much work of this nature has been carried out, very little quantitative work has been attempted, and, in view of its importance from the physiological point of view, it seemed worth while attempting to determine what fatty acids were passed into the milk and under what conditions.

The general plan of the experiment was to give to cows which were under strictly controlled conditions, a basal diet for a sufficiently long period to cause the butter fat to be of something approaching constant composition; at the end of this period, various oils were added to the diet, and after a further period on the basal diet when no oil was given, another oil was administered; after which the cows were put to pasture. The basal diet chosen was one which was as free from fat as possible, in order that the effect of the administration of the oils at a later date on the composition of the butter fat might be the more marked. At intervals samples of butter made from the milk of each cow were examined for the usual analytical values: it was planned to convert the acids of the various butter fats into their methyl esters and to attempt a quantitative separation by a fractional distillation of these esters after the end of the experiment.

PARTICULARS OF THE COWS USED.—The cows chosen for the experiment were dairy shorthorns selected from the herd of the National Institute for Research in Dairying at Reading.

TABLE I.

Cow.	No. of calf.	Last calved.	Condition.
Lily ..	1	17/11/22	Fairly good.
Lucy ..	1	4/12/22	Good.
Scarlet ..	3	8/11/22	Good.
Fillpail II. ..	7	5/1/23	Fairly good.

The cows, all of which had recently calved, were placed in well-lighted stalls at various times between the middle of December and the beginning of January, and were given the basal diet up to February 26th.

COMPOSITION OF THE BASAL DIET.—The basal diet consisted of mangolds, hay and concentrates, the latter made up of equal parts of maize gluten, crushed wheat and soya bean meal, the last having had its fat commercially extracted. The daily ration of each cow varied little from the following:—Mangolds, 60 lbs.; hay, 15 lbs.; concentrates, 9 lbs. (Full details are given in Table 2.)

FAT INTAKE ON THE BASAL DIET.—The percentage of ether-soluble matter in the dried foods was found to be:—Mangold, 0·1; hay, 2·2; maize gluten, 2·2; soya bean, 1·2; and crushed wheat, 1·7 per cent. (dry substance). A half day's ration, after careful drying and powdering, was extracted to remove fat, and yielded 66 grms. of a heavy green oil, having a saponification value of 169·5, iodine value 118·4 and containing 18·75 per cent. of unsaponifiable matter. A table is given later in which the average amount of ether-soluble matter, which can be regarded as saponifiable glycerides, received daily by each cow and worked out over weekly averages for each period, is given.

Throughout the experiment until the final period of grass-feeding, the cows were kept in stall, being only allowed out for a period of fifteen minutes every morning into a clean yard. Records of milk-yield were made at both morning and evening milkings, and samples of every milk were submitted for determination of the percentages of fat and total solids and specific gravity. The food consumption of each cow was measured daily, and close observations of the general conditions of the animals were maintained.

At intervals during the experiment, samples of butter were examined for the usual analytical values and the vitamin concentration, and were then stored at 0° C. in the dark for the quantitative work which it was hoped to carry out at a later stage. Care was taken that samples were always taken at the beginning and end of each period, and intermediately.

PROGRESS OF THE EXPERIMENT.—The duration of the experiment can be conveniently divided into five periods.

Period.	Date.	Diet.
1	12/12/22–26/2/23	Basal: Concentrates, roots, meadow hay.
2	27/2/23 –14/3/23	Basal diet as above + daily doses of coconut or arachis oil.
3	15/2/23 –16/4/23	Basal diet: Concentrates, roots, seed hay.
4	17/4/23 –17/5/23	Basal diet + cod-liver oil.
5	18/5/23 –to end.	Pasture.

Unfortunately, one of the cows, Fillpail II., died suddenly in the early part of period 2. Careful post-mortem examination failed to reveal the cause of death; the other cow receiving coconut oil went off her feed, but her appetite returned rapidly on the cessation of giving this oil.

METHOD OF ADMINISTERING THE OILS.—The oils were given in amounts beginning at 2 ozs. per day and rising to 8 ozs. in small increments, as the cows became accustomed to them. During the oil periods the total weight in lbs. of oil given to each cow was:

	PERIOD 2.			PERIOD 4.		
Oil.	Lily	Lucy	Scarlet	Lily.	Lucy.	Scarlet.
Arachis	9·5	9·5	—	—	—	—
Coconut	—	—	9·5	—	—	—
Cod-liver	—	—	—	10·75	10·75	10·75

The oils were given at the morning feed at first, and later, when larger doses were being given, in two portions at morning and evening feeds. They were mixed with the concentrates and were readily taken by the cows.

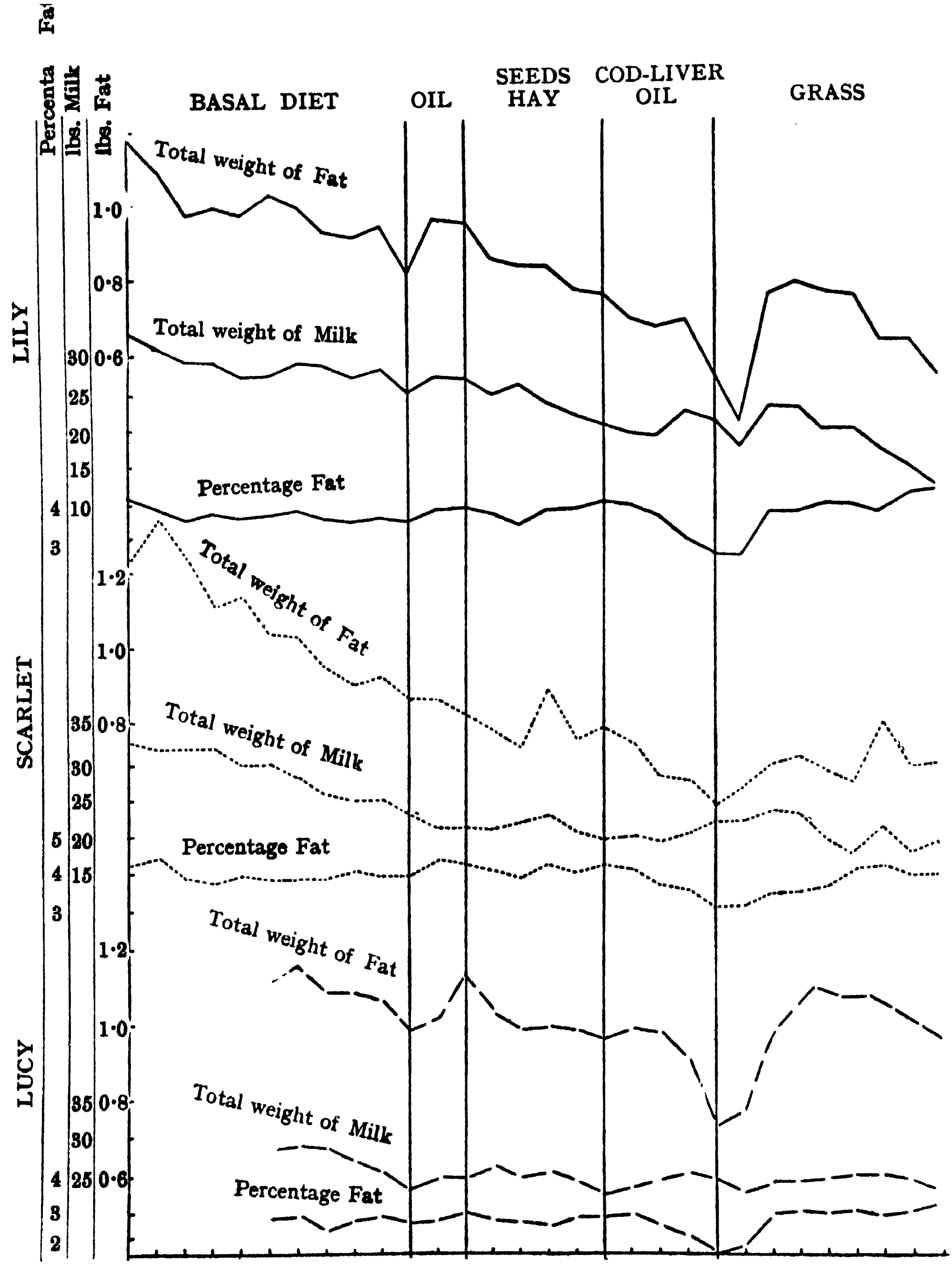

Percentage Fat
lbs. Milk
lbs. Fat
BASAL DIET
OIL
SEEDS
HAY
COD-LIVER
OIL
GRASS
LILY
Total weight of Fat
Total weight of Milk
Percentage Fat
SCARLET
Total weight of Fat
Total weight of Milk
Percentage Fat
LUCY
Total weight of Fat
Total weight of Milk
Percentage Fat
1·0
0·8
0·6
1·2
1·0
0·8
1·2
1·0
0·8
0·6
30
25
20
15
10
35
30
25
20
15
35
30
25
4
3
5
4
3
4
3
2
<— Four Weeks —>

FIG. 1.

The food consumption of the animals was satisfactory throughout the experiment. Such small variations were encountered that the average figures for each period may be accepted as representing the daily intake.

TABLE 2.

Average daily diet in lbs. during the various periods.

Period.	Date.	Diet.	Lily.	Lucy.	Scarlet.
1	12/12/22–26/2/23	Mangolds	52·7	51·6	50·5
		Meadow hay	15·9	15·7	16
		Concentrates	9·1	11·1	11·2
2	27/2/23 –14/3/23	Mangolds	60	58	58
		Hay	15	15	15
		Concentrates (and arachis or coconut oil)	9	12	8·4
3	15/3/23 –16/4/23	Mangolds	60	60	60
		Hay	15	15	15
		Concentrates	7·6	12	8·3
4	17/4/23 –17/5/23	Mangolds	60	60	60
		Hay	15	15	15
		Concentrates (+cod-liver oil)	8	12	8
5	18/5/23 –	Pasture.			

In the final period all three cows were let out to pasture, which was in very good condition; the administration of cod-liver oil was discontinued, except in the case of Scarlet, which continued to receive ½ lb. of oil per day for a time.

RESULTS:—1. YIELD OF MILK AND MILK FAT. Daily records of these values were carefully taken, but for the purpose of simplifying the charts by eliminating minor fluctuations, we give, in Fig. 1, the average daily weight of milk and of fat and also of the percentage of fat in the milk for each cow over periods of one week. In general, it may be said that the curves given in Fig. 1, resemble the lactation curves of normal animals, but there is, in the case of all three cows, a definite fall in the percentage fat during the period in which supplements of cod-liver oil were given. This fall was promptly made good as soon as the cows were put out to pasture in period 5, even in the case of Scarlet, which received cod-liver oil for a time after she had been put out to grass. We are not prepared at this stage of our enquiries to give an opinion on the fall in fat content of the milk during the cod-liver oil period (it would appear to be of an order definitely greater than that which may occur during the late stages of lactation) and further experiments will be necessary to determine whether cod-liver oil in relatively large doses can exert an inhibitive action on the amount of milk-fat formed. No such effect was noted in last year's experiment already reported (Drummond, Coward, Zilva, Golding and Mackintosh, 1923), but in these cases smaller doses of ½ to 4 ozs. of oil were administered. It will be recalled that in Hopkins' experiment on goats (1920) the supplementing of a ration of mixed cereals, hay and mangolds with green food, caused a bigger secretion of milk, but did not influence the proportion of total solids or fat.

The following table shows the average daily weight of fat in the milk per day, the percentage of fat and the fat ingested by the cows in the food over the five periods, but it may be unwise to lay too much importance on figures which are averaged over periods differing considerably in length.

	Lily.			Lucy.			Scarlet.		
Period.	Fat in Milk. lbs. per day.	Fat in Food. lbs. per day.	Fat in Milk. Per Cent.	Fat in Milk. lbs. per day.	Fat in Food. lbs. per day.	Fat in Milk. Per Cent.	Fat in Milk. lbs. per day.	Fat in Foods. lbs. per day.	Fat in. Milk. Per Cent.
1	0·95	0·37	3·7	1·07	0·39	3·9	1·08	0·39	3·7
2	0·95	0·95 (arachis oil)	3·8	0·84	0·94 (arachis oil)	4·2	1·06	0·98 (coconut oil)	3·8
3	0·80	0·34	3·7	0·78	0·35	4·0	0·99	0·39	3·6
4	0·60	0·69 (cod-liver oil)	3·3	0·65	0·60 (cod-liver oil)	3·5	0·86	0·74 (cod-liver oil)	3·3
5	0·68	—	3·7	0·69	—	3·6	1·08	—	3·8

It will be seen that the fall in the percentage and total fat is distinctly more marked in period 4 than in any other period.

2. QUALITY OF THE MILKS AND BUTTERS.—All the samples of milk from which butters were made were carefully observed during churning. No sample of milk was found to possess a "fishy" flavour, but the considerable abnormalities which were noticed in certain samples during butter-making are dealt with in the sections on the chemical analysis of the butter-fats. During the periods of oil-feeding, certain of the butters tended to be a little oily, but this was chiefly when the animals were retaining the vegetable oils in period 2. In period 4, no "taint" was noticed in the butters, even when the cows were receiving as much as ½ lb. of cod-liver oil per day each.

3. VITAMIN CONTENT OF THE BUTTERS. This question forms the subject of another examination (*J. Agric. Sci.* 1924). The results may be summarised as follows:—

(*a*) The typical winter ration of concentrates, roots and hay, may be adequate to maintain the vitamin *A* value of the milk-fat for considerable periods of time, provided that at least one of its components supplies adequate amounts of that dietary principle. Fresh green meadow hays are in this respect greatly superior to dry, brown, seeds hays.

(*b*) The addition of cod-liver oil to a winter ration deficient in vitamin *A* will induce a sharp rise in the vitamin *A* concentration of milk-fat. No such effect is seen when oils deficient in this dietary accessory (coconut and arachis oils) are given.

4. CHEMICAL ANALYSIS.—

(a) *Reichert-Wollny Values.*

Period.	Date.	Diet.	Lily.	Lucy.	Scarlet.
1	11/12/22	Basal	34·4	—	—
	18/12/22		31·6	—	30·2
	23/1/23		32·9	31·2	28·4
	19/2/23		29·8	28·7	27·5
2	5/3/23	Basal oil (Scarlet, coconut; Lily, Lucy, arachis)	31·5	32·2	28·5
	15/3/23		29·6	30·9	25·2
3	9/4/23	Basal (seeds hay)	29·3	31·2	29·4
4	30/4/23	Basal (cod-liver oil)	30·0	31·4	30·2
	13/5/23		24·9	23·5	26·5
5	29/5/23	At grass, Scarlet received cod-liver oil until	23·8	20·9	21·9
	19/6/23		24·1	23·0	23·4
	9/7/23		20·1	21·5	21·5

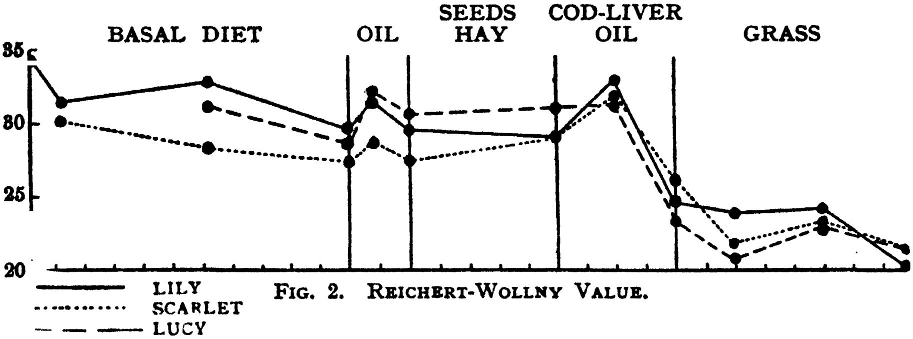


FIG. 2. REICHERT-WOLLNY VALUE.

Fig. 2 gives the fluctuations of the Reichert-Wollny value of the butter fats from each individual cow throughout the experiment. The average value for normal butter fat is 28·5; for coconut oil, 8·5; cod-liver oil and arachis oil do not contain water-soluble volatile acids. The curves show a general fall in the Reichert-Wollny values throughout the seven months of the experiment. It will be observed that the values were higher at the beginning than are the average values, and this is to be expected, as the cows had calved just prior to the commencement of the experiment. There is no effect apparent when the oils were given in period 2, at the end of which the values differ but little from those at the beginning, and the general level is maintained at 30 through the third period, when no oil was given. It is to be expected that a decided diminution in the amount of volatile soluble acids would occur in period 4 when cod-liver oil was given for 4½ weeks. This is borne out by curves which show marked similarity of slope; at the end of this period the average value was below the limit of 24, which is usually taken for unadulterated butter fat. It is curious to note that the Reichert-Wollny values tended to rise in the middle of the cod-liver oil period, and this seems to indicate that there are unknown factors in this type of experiment which are uncontrolled. Further, the fall in the values during this period from 30 to 23 was continued for a time after the cows were put out to grass, although it was less rapid at this stage. There was no real recovery in the amount of volatile acids in the butter fat, even after the cows had been out at grass for 8 weeks. This may suggest that the fall during the cod-liver oil period was due to the advancement of the lactation period. It was shown by Nilson for Swedish butter that the Reichert-Wollny value of the butter fat from a group of cows fell from 33·4 in the first month of lactation to 25·4 in the fourteenth month. Many other workers report the big fall in the volatile acids at the end of the lactation period, but it is clear that the fall in our experiment from 33 in the first month to 22 in the seventh is of considerably greater magnitude, and is presumably due to the administration of an oil devoid of those acids. This is confirmed by the other analytical data.

(*b*) *Saponification Values.*

Period.	Lily.	Lucy.	Scarlet.
1	232	—	—
	227·7	—	232
	234·3	230·5	238·5
	236·7	228·8	228·8
2	231·6	234·3	235·6
	222·2	229·3	229
3	237	238·4	235·8
4	233·8	229·2	229·2
	221·9	219·5	222·5
5	219·0	217·3	216·4
	218·9	216·8	217·3
	218·1	219	220·3

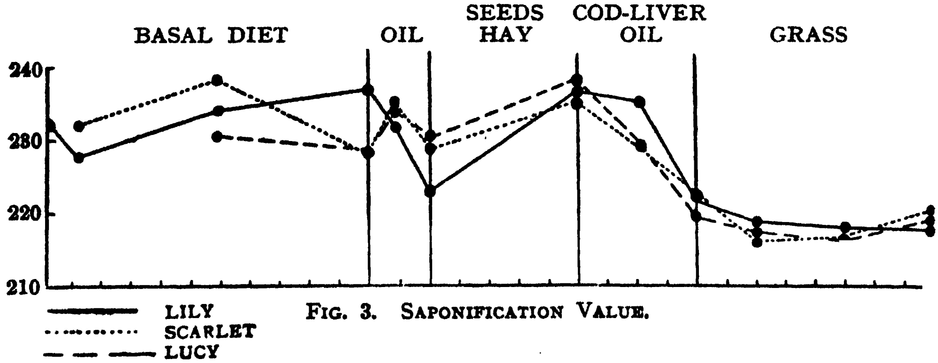


FIG. 3. SAPONIFICATION VALUE.

The average saponification value of normal butter fat may be taken as 227; of coconut oil, 257; arachis oil, 193; cod-liver oil, 185. Fig. 3 shews that the saponification values at the beginning of the experiment were higher than normal, following the Reichert-Wollny value. In the case of one cow only is there an appreciable fall in the saponification value of the butter fat at the end of the arachis oil feeding, which is in agreement with the results obtained from the iodine values. There is no rise in the value from the butter fat of the cow receiving coconut oil at the end of the period, but a distinct rise in the middle, which is reflected in the rise in iodine value and refractive index of the butter concerned. An average rise of about 7 in the saponification values had occurred at the end of period 3 during which the meadow hay of the diet had been replaced by seeds hay and when no oil was being given. Immediately following the administration of cod-liver oil in period 4, the values begin to fall, and at the end of this period, 4½ weeks later, they have reached the low figure of 221. There is no marked rise in the values when the cows return to grass, but at the end of eight weeks they are tending to increase. This failure to return to normal values is also seen in the Reichert-Wollny and iodine values, and the refractive index.

(c) *Iodine Values.*

Period.	Lily.	Lucy.	Scarlet.
1	38·6	—	—
	40·3	—	39·7
	31·8	25·5	30·1
	28·7	32·2	31·0
2	36·4	33·2	27·4
	39·9	40·8	33·4
3	28·0	25·6	27·0
4	41·8	38·6	35·0
	55·6	52·1	46·6
5	46·7	46·8	51·0
	45·3	45·2	44·5
	47·1	46·3	46·9

The average iodine value for normal butter fat is 33; of coconut oil 8·5, and of arachis and cod-liver oils 90 and 170 respectively; hence the effect to be anticipated, if direct passage of the fatty acids occurs, is a decrease in the values of the butters from cows fed on coconut oil and a corresponding increase in those from cows fed on arachis oil; cod-liver oil should yield butters shewing abnormally high iodine values. Fig. 4 gives curves shewing the variations in iodine values throughout the experiment. In one case only of the cows receiving arachis oil is there a distinct rise, while the butter from the cow receiving coconut oil had an iodine value at the end of the period as high as that at the beginning. This curious result is confirmed by the refractive indices which are given in Fig. 5. The iodine values show a slight fall during the period when the cows were receiving seeds hay in the basal diet, but from the commencement of the cod-liver oil period there is a sharp rise, which is maintained throughout, from an average figure of 26·9 at the commencement to 51·4 at the end. There was a general fall in the values after the cows were turned out to pasture, save that the iodine value of the butter fat from Scarlet, which received further cod-liver oil for a period while at grass, shows a further rise until administration of the oil ceased. It will be observed that even at the end of the 8 weeks' grass-feeding period the iodine values tend to remain high.

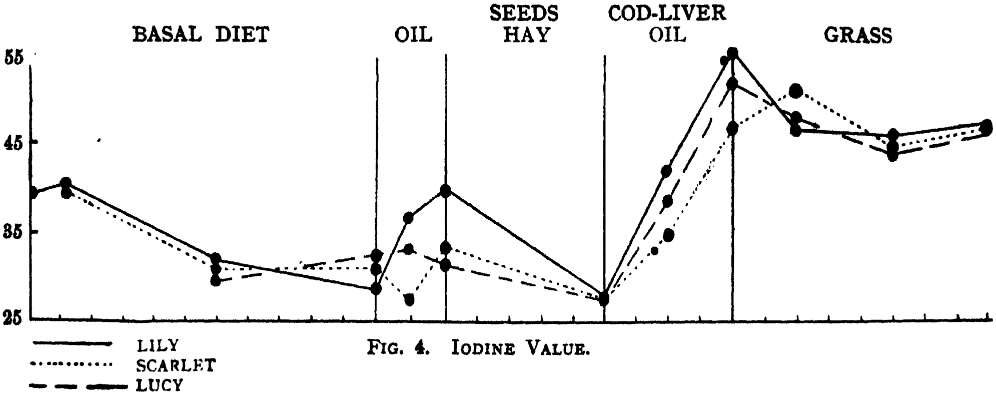


FIG. 4. IODINE VALUE.

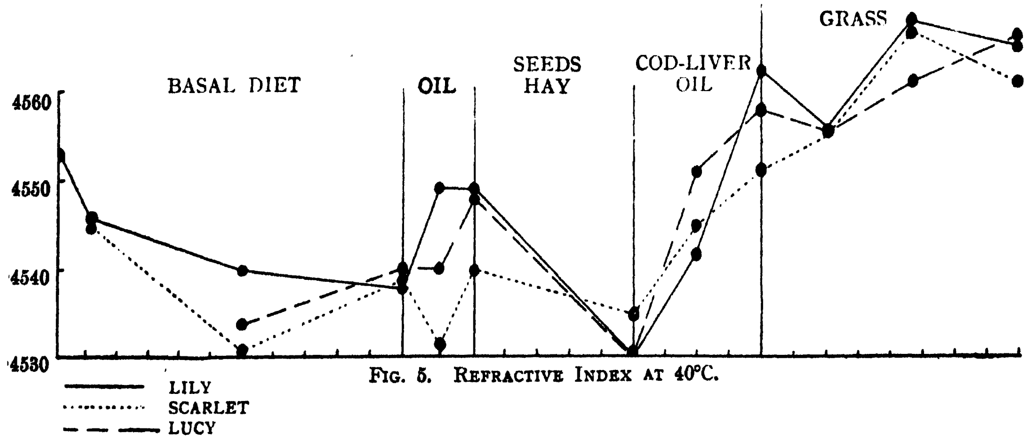


FIG. 5. REFRACTIVE INDEX AT 40°C.

(*d*) *Refractive Index.* (40° C.)

Period.	Lily.	Lucy.	Scarlet.
1	1·4553	—	—
	1·4545	—	1·4545
	40	1·4534	31
	38	40	39
2	49	40	32
	49	48	40
3	31	30	35
4	42	51	45
	62	58	51
5	56	56	55
	61	68	67
	65	66	61

The refractive index of normal butter fat is 1·4548; of coconut oil, 1·4493; arachis oil, 1·4745; cod-liver oil, 1·4723. In general, the refractive index follows the same course as degree of unsaturation, and hence there should be a similarity between the curves shewing refractive indices and iodine values. The curves of Fig. 5 resemble those of Fig. 4. There is a rise from an average of 1·4540 to

1·4548 in the refractive indices of the butters from the cows receiving arachis oil; but a slightly higher value than that of the butter at the beginning of the period obtains in that from the cow receiving coconut oil. There is a decided fall in all the values in period 3 to about 1·4535. The administration of cod-liver oil caused a marked rise which continued throughout the period to a value 1·4556. A temporary fall then occurred after the return of the cows to grass, but Scarlet, which was still receiving oil, yielded a butter of still higher refractive index. This temporary fall is followed by a rise until the end of the experiment, although by this time the iodine values had fallen somewhat.

Some explanation of this may be found in the fact that as the amount of water-soluble volatile acids decreases as the period of lactation progresses, the refractive index tends to rise. Against this is the fact that increase in iodine value causes an increase in refractive index, which is much more marked than the effect due to the variation in the Reichert-Wollny values. These latter had fallen to the low figure of 22·2 from 24·9, and this would therefore cause a rise in the refractive index, though the rise would not be expected to be such as to counterbalance the fall caused by the decrease of the iodine values from 51·4 to 46·7.

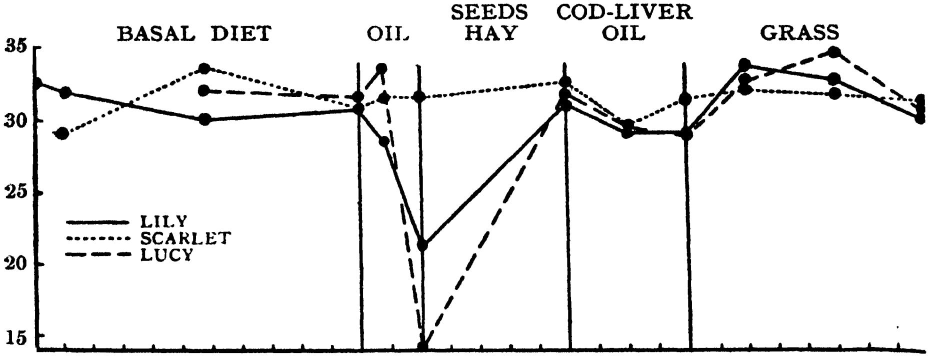


FIG. 6. MELTING POINT.

Melting Point.

Period.	Lily. °C.	Lucy. °C.	Scarlet. °C.
1	32·5	—	—
	31·5	—	29·0
	30·0	32·0	33·0
	30·5	31·5	30·5
2	28·5	33·5	31·5
	21·0	14·0	31·5
3	31	31·5	32·5
4	29	29·5	29·5
	28·7	28·7	31·2
5	33·5	32·5	32·0
	32·5	34·5	31·5
	30·0	30·0	31·0

The melting point of butter fat is 29° to 33° C. Very abnormal melting points were found in the butter from the cows receiving arachis oil, falling as low as 14·5° C. This result is curious, for the other analytical values obtained, though abnormal, do not suggest so great an alteration in the composition of the butter fat. That abnormal effects on melting points can be obtained is shewn by the fact that the use of cotton seed cake is reputed to cause a rise in melting point. The use of cod-liver oil in our experiment had very little effect, even though the cows received 10·75 lbs. each in the course of 4½ weeks; the coconut oil did not cause any appreciable alteration in the melting point of the butter.

DISCUSSION.—Consideration of the curves indicates that no very marked effect, apart from the abnormally low melting points just referred to, on the characteristics of the butter fat resulted during the period when the basal diet was supplemented by coconut and arachis oils. This is a little surprising, in view of the definite change induced by the cod-liver oil feeding, although the period during which cod-liver oil was being given was more than twice as long as that of the other oils. It was to be expected that even though the effect due to coconut oil, which bears some similarity in composition to butter fat, was not marked, more definite evidence would have been obtained from the administration of arachis oil, which gives analytical values resembling those of cod-liver oil.

During the cod-liver oil period the analytical values all varied markedly in the direction indicating considerable passage of the oil or of some of its constituents into the milk. The slopes of the curves of all the values are very similar, and the close resemblance over the whole experiment is striking. It is of interest to note that the alterations in the analytical values tend to be maintained throughout the period of 8 weeks when the cows were put out to pasture and the giving of oils had ceased; reference has already been made to the sharp rise which occurred in the yield of fat.

It is difficult to draw conclusions as to the passage of oils into the butter from such figures as are provided by the analytical values. It will be seen that quite marked variations occurred, even in the first period of the experiment, when the cows were receiving the fixed basal diet only. Hence there is no justification for taking the value of any given constant at the beginning of a period of oil feeding as that which would have been maintained had no oil feeding occurred, for the variation, even when the animals were on a fixed diet, demonstrates that there are factors which cannot be controlled. Figures representing the difference between the constants for any analytical value at the beginning and end of an oil period are valueless, unless they show a considerable percentage difference from those at the commencement. With the stipulation that these differences give only an approximate idea of the change in composition induced by the oil feeding, we may consider them for the cod-liver oil period only; they are not sufficiently definite during the arachis and coconut oil period to be of value. The following table gives the average values of the constants from the butter-fats of all three cows at the beginning and end of the cod-liver oil period.

Reichert Wollny value	Saponification value.	Iodine value.	Refractive index.
29·6–24·9	237·1–221·3	26·9–51·4	1·4535–1·4556

If we assume that the modification of the butter fat indicated by these figures was due to passage of the complete oil into the milk, and that there was no preferential passage of any individual constituent, we may calculate what quantity of cod-liver oil added to butter fat, of the average composition possessed by those at the commencement of period 4, would yield a butter fat having the analytical values given above at the end. This gives 12 per cent. in the case of the iodine value, 30 per cent. for the saponification value, 7 per cent. for the Reichert-Wollny value, and 11 per cent. for the refractive index. Three of these figures agree well, but it is to be emphasised that no conclusion can be drawn from these calculations as to whether individual constituents of the oil have been preferentially passed into the fat, although they serve to bring out the change caused by the cod-liver oil feeding.

A further point of interest is that the amount of fat in the milk produced by the cows per day during the time when they were receiving the basal ration only, without oil supplement, was from two to two and half times the amount actually present in the diet, yet when the amount of cod-liver oil given per day was about 75 per cent. of the amount of fat yielded per day, only about 12 per cent. of this oil appeared in the milk.

As remarked above, it was hoped by careful fractionation of the methyl or ethyl esters of the fatty acids of the butters to obtain more definite information regarding the passage of fatty acids from the food into the milk, particularly in the case of acids which are present in arachis and cod-liver oil but normally absent from butter fat.

FRACTIONATION OF METHYL ESTERS.—The method of fractional distillation of the esters from the fatty acids has been employed in the case of butter fat by numerous investigators, including Smedley (1911–1912), Crowther and Hynd (1917), Holland and Buckley (1918), Holland and others (1923), and with other oils by Bull (1906), Elsdon (1913). The methyl esters were prepared by passing dry hydrogen chloride gas into a solution of the dry fatty acids obtained in the usual manner in methyl alcohol boiling under a reflux condenser; the methyl alcohol had been allowed to stand over fresh quicklime for several days and had been redistilled from lime twice. The esters were removed by means of a separating funnel, more methyl alcohol added, and further separation made after again heating under a reflux condenser with hydrogen chloride. The united esters were then dissolved in ether and washed with successive small quantities of distilled water, to remove the dissolved hydrochloric acid, until the washings did not affect blue litmus. The ether solution was then allowed to stand overnight in contact with anhydrous sodium sulphate, and the clear yellow solution filtered from the sodium sulphate, which was then washed with ether distilled from sodium. The combined filtrate and washings were freed from the bulk of the ether by heating on a water bath to 100° C., the flask and its contents were then heated to 130° C. in order to remove the last traces of ether. This method of preparation resulted, of course, in the loss of the greater part of the lower esters. No effort was made to avoid this in this preliminary work, because it was felt that the real difficulty would be encountered, not in the separation of the esters of the lower acids, but in obtaining those of the acids with more than 6 carbon atoms in a pure state by fractionation, and if the quantitative separation of the lower acids later proved impossible, much information could be obtained by carrying out the Reichert-Meissl, Polenske and Kirschner determinations.

The esters were then submitted to fractional distillation in a double-necked flask, one limb of which contained a five-pear fractionating column. The distillation was carried out at a pressure of 15 mm. or thereabouts, such as is usually given by water-pumps, and every effort was made to keep the pressure as constant as possible. The whole of the upper part of the flask was well lagged with a thick layer of tightly packed asbestos, and the distillate was received into a vacuum receiver of the Bruhl type, containing twelve tubes, each of 15 c.c. capacity. It was thus possible to receive at a maximum 180 c.c. of esters without any intermission in the heating or increase in pressure. An approximate idea of the progress of the fractionation was obtained by determination of the iodine value of the individual fractions. At the end of the first fractionation the contents of the tubes which contained esters boiling at points on either side of the boiling point of that of each pure ester were mixed, and a second fractionation then carried out. At the end of this fractionation a similar procedure was adopted, and this was repeated four times in all.

Typical of the fractionations may be quoted the following, which gives the results obtained during the first distillation of 150 grms. of methyl esters:

Fraction.	Pressure.	Temperature °C.	Iodine value.
1	17 mm.	70–100	0
2		101–130	10·2
3		131–160	20·3
4		161–163	22·7
5		164–175	23·8
6		176–190	32·9
7	15 mm.	191–196	35·1
8		197–200	40
9		201–204	40·1
10		205–209	43
11		210–213	46·5
12		214–218	50·8
13		undistilled residue	61·7

The figures obtained for the iodine value after three more distillations of this material were not substantially different from those quoted in the foregoing list.

Various modifications were made in the flask by alteration of the type and length of the fractionating column, but without any real improvement in the purity of the fractions, as judged by their iodine values. It was considered, therefore, that it would be impossible to obtain by fractionation pure esters which could be weighed as individual substances, and, after much further work, we were reluctantly forced to adopt the opinion that, as a method of quantitative separation of the fatty acids of butter fat, the process is of little practical use. The method employed by Holland and others (1923) is substantially that published by Holland and Buckley in 1918, and very similar to that described by Crowther and Hynd (1917). The former report the results of a series of experiments on the composition of butter fat from cows at various stages of lactation, and on different diets. They distil the ethyl esters of the fatty acids of butter fat at atmospheric pressure once only; they receive into flasks six fractions, and, from the weights of the contents and their saponification and iodine values, they claim to be able to give an analysis of butter fat to three decimal places per cent. Their calculations are based on the assumption that each fraction will contain oleic ester and the esters of two succeeding members of the saturated series of esters and no others. Thus the first fraction will contain butyric, caproic and oleic esters, the second caproic, caprylic and oleic esters, and so on. Each saturated ester will thus appear partly in each of two successive fractions and no others. If this be admitted, and also the assumption that oleic ester is the only unsaturated ester present, then a determination of iodine value will provide a means of calculating the amount of oleic ester in each fraction. This weight, on subtraction from the weight of the fraction, leaves the combined weight of the two saturated esters, and, from a determination of the saponification value, the weight of each of these can be found algebraically. From our own experience we think that objection should be taken to some points of this work. Thus the iodine and saponification values are given to three decimal places, which would imply an accuracy in these determinations which we do not believe it possible to attain. Further, the temperature which is reached in the distillation of ethyl esters at atmospheric pressure (the last fraction is received at a temperature of 365° C., which would probably mean one of over 400° C. in the flask) is such that considerable decomposition is bound to occur, for we found that in a similar distillation of the esters from palm-kernel oil, considerable loss occurred on account of decomposition, and even in distillation at 15 mm. pressure, the loss on this account is not negligible. Nor does it appear wise to weigh each fraction to four places of decimals, in view of the fact that it is collected merely by means of a flask exposed to air and standing at the end of a condenser. On these grounds alone it would appear that the authors have ascribed to their work a degree of accuracy which cannot be reached in work of this type, and hence there is no justification for their expression of their results of the analysis of the fatty acids of butter fat to three decimal places per cent. when such things as decomposition, presence of unsaponifiable matter, etc., have been neglected.

The question whether each fraction will contain two saturated esters and oleic ester only is of much greater importance, and the whole method employed by these workers is dependent on this being true. Crowther and Hynd (1917) have reported that this is borne out by experiment. In a study of the question, they made an artificial mixture of the methyl esters corresponding to an approximate composition which such esters prepared from true butter fat would have, distilled this mixture up to 150° C. at atmospheric pressure, taking adequate precautions to ensure no loss of the lowest esters on account of their volatility, and then the remainder in vacuum. This fractionation was repeated three times, and, working on the principle that each fraction contained two successive saturated esters and oleic ester and no others, the analysis found by this method corresponded extremely closely to the actual composition of the mixture of esters as made up. Their recovery was so high as 95 per cent. Thus the method would appear valid, but it seems to us fundamentally unsound to assume that the vapour pressure curves of the saturated esters should differ so greatly from that of oleic ester that each of the former substances would appear in two successive fractions and no others. Surely, if it is reasonable to expect that oleic ester appears in the lower fractions, palmitic and stearic esters, etc., will tend to behave in somewhat similar manner, but the inability to detect them in so ready a manner as is given by the iodine value in the case of oleic ester is due to their lack of specific chemical properties. Further, even if the method be accepted, it appears very difficult to decide at what point each stage of the fractionation should come. Crowther and Hynd say that they found it necessary to fractionate three times, whilst Holland and Buckley's statement that "the object was to secure fractions that did not contain more than two esters in addition to oleic ester, and, furthermore, adjacent fractions should contain approximately half the saturated ester appearing in each," and the fact that they fractionate once only, would seem to introduce an element of chance which renders the method of no value for quantitative work.

It is to be pointed out that, because the saponification value of any fraction of the working, after allowance for oleic ester, lies between those of two adjacent esters, there is little, if any, justification for saying that these two esters alone are present, as it would be possible to make many mixtures of esters having any given saponification value and containing varying quantities of several esters.

The conclusion to be drawn from our experience with fractionation of the esters of the butter fat fatty acids is that, as an exact quantitative method, it is of little value. Furthermore, we are inclined to question whether the many published figures for the composition of butter fat often given to two or three places of decimals per cent., which are based on the ester method, are of more value than mere approximations. This method would appear to be of the same order of accuracy as E. Fischer's ester method for the analysis of proteins; that is, it can yield very valuable information if its limitations are recognised.

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DISCUSSION.

Mr. E. M. HAWKINS said he thought it was a pity some normal cow had not been put alongside the test cows, for it was well-known that the Reichert value of the butter from cows' milk towards the end of the lactation period sometimes ran very low; he suggested that the drop at the end of the curve might be due to this.

Mr. E. R. BOLTON, having complimented the authors upon the utility of their work, raised the question as to how far the vitamin strength *per se* of an oil affected the fatty composition of the butter, and as to whether the same given oil (rich in vitamin, or free from vitamin) would have a similar effect.

Capt. J. GOLDING, commenting on the paper, emphasised the practical importance of these discoveries to dairy farmers. The winter feeding of dairy cows required revision in the light of these newly discovered facts. The advantages of meadow hay for winter feeding as a better source of vitamin *A* than seeds hay was a point of the greatest practical importance, and might well be a factor in maintaining the food value of our winter milk. Many American workers had found a negative calcium balance when using sun-dried alfalfa hay.

Mr. G. R. THOMPSON enquired whether any attention had been given to any relation between the specific gravity and the melting point of the fats.

Dr. J. C. DRUMMOND, in reply, said that they had brought the paper before the Society very largely because they had felt that their six months' effort had been wasted. According to text books it was a relatively simple matter to get a satisfactory fractionation of methyl esters.

The chart which had been thrown on the screen giving the figures obtained by the American workers had caused amusement, but those workers were on the staff of an important Experimental Station. Not only were their figures published in a responsible journal, but they were reproduced in the text-books, and he felt it was time these inaccuracies were exposed. Regarding the point raised by Mr. Bolton: this had not actually been studied. As far as they knew, vitamin *A* had no direct influence on the composition of the milk-fat. In reply to Mr. G. R. Thomson's question, they had not observed any correlation between the specific gravity and the melting point of the butter. Butters of cows fed with cod-liver oil were tested for palatability, and, although the butter tended to be rather insipid, it had no objectionable flavour.

The Composition and Decomposition of Eggs

BY R. T. THOMSON AND JAMES SORLEY.

(*Read at the Meeting, March* 5, 1924.)

COMPOSITION OF EGGS.—During recent years we have made a large number of analyses of eggs, and in our experience there is reason for revision of some results which have been published. This we propose to do in the present paper, as well as to record some further data.

The method we adopt for the estimation of fat or oil in whole egg (edible portion) or yolk is as follows:

A weighed quantity of the sample is dried at 100° C. and the residue macerated with a flat-headed rod in a basin, using methylated ether as the solvent. The ethereal solution is decanted through a small filter, and the non-fatty solids treated repeatedly with small quantities of ether until the fat is completely extracted, the fat being recovered in the usual way. Jean states that different solvents give varying percentages. His figures for dried yolk are as follows:

	Fat extracted Per Cent.
By petroleum spirit	48·24
By ether	50·83
By chloroform	57·66

We, on the other hand, find that extraction is complete, whichever of the three solvents is used.

When petroleum spirit is used as the solvent it is advisable to have the sample absolutely dry, but with ether the drying need not be perfect. In the case of chloroform the addition of sand or other inert substance is necessary, as otherwise the non-fatty matter floats in the solvent, thus rendering extraction very difficult.

We may note here that in extracting yolks or whole eggs which have been preserved with 1 to 2 per cent. of boric acid a little of that acid, amounting usually to about 0·15 per cent., is extracted, and is obtained in the fat. It can, however, be expelled by adding methyl alcohol to the fat, when the boric acid is vaporised as methyl borate at 100° C. Chloroform only extracts traces of boric acid.

Further tests were also made with the object of extracting the fat with the solvents in a separator, the yolk being previously mixed with about ten times its weight of water. The extraction with petroleum spirit or ether was troublesome, and with chloroform practically impossible, owing to emulsification taking place. It was found that several treatments with ether extracted all the fat, but that petroleum spirit only yielded a very small percentage. Samples of hen's yolk and duck's yolk gave the following results:

Fat extracted	Hen's Yolk. Per Cent.	Duck's Yolk. Per Cent.
By ether	31·10	36·20
By petroleum spirit	0·48	0·43

Chloroform applied to the undiluted yolk extracts the fat fairly well.

We have made various analyses of hens' and ducks' eggs, but the results for fat, proteins, etc., come within the limits recorded in various works. Our aim was chiefly concerned with the change which takes place in the fat of yolks when kept for a period, and the percentage of free fatty acid in a perfectly fresh egg was of importance A hen's egg, the analysis of which was commenced one hour after laying, was tested, with the following results:

	Yolk. Per Cent.	White. Per Cent.
Fat	34·63	trace
Other organic matters	16·97	11·04
Mineral matter (ash)	1·49	0·83
Water	46·91	88·13
	100·00	100·00
Free fatty acids (as oleic acid)	0·59	—
Free fatty acids in the fat	1·72	—
Acid value	3·42	—
Unsaponifiable matter in the fat	4·28	—

This yolk was exceptionally rich in fat, but other samples did not show above 32 per cent. The yolk in this egg formed 38·4 per cent. of the whole edible portion, but in other eggs the amount of yolk ranged between that figure and 43·3 per cent.

The free fatty acids are low, as was to be expected, but the unsaponifiable matter is much higher than any recorded results. For this item a figure as low as 0·2 per cent. is stated by Vignon and Meunieur, whilst other observers give 1·5 and 3·0 per cent. of cholesterol, which forms part of the unsaponifiable matter. For the fat of ducks' eggs, Vignon and Meunieur give 2·7 per cent. of unsaponifiable matter.

The estimation of unsaponifiable matter was made by saponifying in the usual way and extracting with methylated ether, which is our usual procedure. In order to determine whether different solvents might give varying results, we extracted the fat from a hen's egg and a duck's egg, and carried out extractions with methylated and petroleum spirit respectively. The results, as well as those for other constants, are recorded below:

	Hen's Egg. Per Cent.	Duck's Egg. Per Cent.
Unsaponifiable matter (petroleum spirit)	0·36	0·28
Unsaponifiable matter (methylated ether)	3·75	4·27
Iodine value (Wijs)	74·73	75·51
Saponification value	183·79	180·90
Acid value	4·47	5·70
Free fatty (oleic) acid, per cent. ..	2·25	2·87
Reichert-Meissl value	0·62	0·27
Polenske value	0·28	0·25

It has been generally assumed that petroleum spirit extracts all the unsaponifiable matter from fats or oils, but this is very far from being the case with egg fat, and it is quite possible that other fats may show the same result. The case mentioned above, where only 0·2 per cent. of unsaponifiable matter was obtained, is probably due to petroleum spirit having been used for the extraction. The iodine value for hen's egg fat is consistent with some recorded figures, but 37·4 has been given for duck's egg fat. The other constants practically agree with the recorded results, which are extremely few. Other results for hen's egg fat were 4·54 and 4·28 per cent., and for duck's egg fat, 4·86 and 4·05 per cent. of unsaponifiable matter.

We have also directed our attention to the constants for fat in Chinese preserved hen's egg yolk, and our results are stated below:

	No. 1. Per Cent.	No. 2. Per Cent.
Unsaponifiable matter (ether) ..	3·64	4·57
Iodine value (Wijs)	78·51	84·20
Saponification value	186·10	184·30
Acid value	10·42	51·88
Free fatty (oleic) acid, per cent. ..	5·24	26·08
Reichert-Meissl value	2·5	0·9
Polenske value	0·4	0·7

Sample No. 1 was in good condition, but No. 2 was about 2 years old, and had decomposed, assuming a thick or pasty condition. Another sample yielded 4·73 per cent. of unsaponifiable matter.

A point of some interest that we have not seen referred to specially is the reaction of yolk and white of egg to indicators. A fresh hen's egg, and one 7 months old, gave the following results:

White.		Hen's Egg (Fresh). Per Cent.	Hen's Egg (7 months' old). Per Cent.
Alkalinity (as NaOH) by methyl orange	..	0·08	0·51
Alkalinity (as NaOH) by phenolphthalein	..	0·02	0·08
Yolk.			
Acidity (as SO_3) by phenolphthalein ..	..	0·15	—

It is impossible to say what the alkalinity of the white, or the acidity of the yolk, is due to, as the effect of proteins and other constituents on the indicators is not known. The yolks appeared to be somewhat acid to methyl orange, but a good end-point was not obtainable. It may be noted that when yolk is treated with a 10 per cent. solution of sodium chloride, the fat extracted with ether, and the vitellin precipitated by addition of water, the filtrate gives practically the same acidity as the original yolk. The above results were for an egg that was certainly newly laid, but higher alkalinity is obtained in the white according to the age of the egg. The acidity of the yolk, except that due to the free fatty acids, does not increase. Ducks' eggs give practically the same results as hens' eggs for alkalinity of the white and acidity of the yolk. The ash of hens' eggs was also tested, with the following results:

White.		In Ash. Per Cent.
Alkalinity (as NaOH) by methyl orange ..	..	6·31
Alkalinity (as NaOH) by phenolphthalein ..	..	2·24
Yolks.		
Acidity (as SO_3) by methyl orange	..	2·65
Acidity (as SO_3) by phenolphthalein ..	..	25·50

The acidity of the yolk ash towards methyl orange indicates that there is free phosphoric acid in it, to the extent of 4·7 per cent. of phosphoric anhydride. Similar results were obtained with ducks' eggs.

DECOMPOSITION OF EGGS.—When a shell egg is kept for a considerable time, decomposition sets in. The white decomposes and the yolk also undergoes decomposition. The former decomposes much more quickly than the latter, but in this paper we have only concerned ourselves with the decomposition of the yolk.

It is not generally known that when the yolk is kept for some time, fermentation sets in and free fatty acids are produced from the fat. The yolk of an egg, tested 1 hour after laying, showed the presence of 1·72 per cent. of free fatty acids (stated as oleic acid) in the fat, whilst after three months the amount of free fatty acids is very little increased. After one year, this figure rises to 3·12 per cent., and at the end of two years to 5·15 per cent. These figures are the average of a number of tests, as we found that the free fatty acids in two eggs laid at the same time varied slightly.

The reason for this decomposition proceeding so slowly is that the yolk is well protected inside the white of egg.

We also made experiments with fresh eggs which were shelled and placed in a bottle with a tight-fitting stopper. After one year the fat was extracted, and the free fatty acids were found to be 85·5 per cent., calculated on the fat.

Further, a hen's egg of uncertain age, but which was unmistakably bad, was found to contain 17·3 per cent. of free fatty acids. The shell of this egg was cracked, and to this cause we attribute this high result.

Experiments were also made with Chinese hens' egg yolks, which are shipped to this country in bulk, without shells.

Two samples were tested with the object of ascertaining how quickly the free fatty acids are produced. The tests gave the following results:

	Free Fatty Acids.	
	Prime Quality. Per Cent.	2nd Grade Quality. Per Cent.
1st test	6·5	12·2
2nd test, after 6 weeks	7·4	13·2
3rd test, after 6 months	11·2	15·9

The above results show that free fatty acids are produced with time, but we were quite unable to find any definite relationship between time and production of free fatty acids. Our tests showed that a preservative such as boric acid slowed down the production of free fatty acids very considerably, but not altogether, as the samples of Chinese yolks tested above, were preserved with boric acid. On one point we were able to gain definite information, namely, that free fatty acids are produced more quickly in the summer than in the winter.

A series of tests with imported yolks showed that when the free fatty acid figure became high, the eggs were practically useless for whisking.

DISCUSSION.

Mr. M. S. SALAMON commented on the large amount of data—convenient for reference—which had been collected in the paper. The authors stated that it was immaterial which solvent was used for the extraction of the fatty matter, but, in his opinion, with petroleum spirit the extraction was by no means complete. The extractions were contrary to the methods employed by earlier workers, but confirmed the known fact that a larger extract was obtained with chloroform than with any other solvent.

The characteristics of chloroform-extracted yolk differed entirely from those of yolk extracted with any other solvent. He thought it a pity the authors had not returned the percentage of nitrogen in the fat, which might possibly have explained the divergence in the results with petroleum spirit and ether. This difference was not found with olive and similar oils, which differed very materially from an oil or fat, such as egg oil, that contained a large percentage of lecithin or basic substances.

Regarding the authors' experiments on fatty acids, the speaker did not think there was any relationship between time and the percentage of fatty acid content; the authors did not appear to have differentiated between the fatty acids produced by decomposition, lipase, moulds or bacteria. Eggs contained different fatty acids which could be detected by differences in the flavour.

Mr. E. HINKS said that he doubted the advisibility of drying the egg alone and extracting the dried residue; if the egg were dried as said, both the drying and subsequent extraction were facilitated. With regard to the unsaponifiable matter, petroleum spirit gave erroneous results with any oil containing much cholesterol, a fact which no doubt accounted for the low figures given by the authors. He agreed with Mr. Salamon's view that, although the authors advise methylated ether for extraction of the oil, their figures shewed that chloroform was the best solvent.

Dr. H. E. COX said he considered the method of drying the original egg as important as the method of extracting it. He had recently read a paper in an American journal in which the extract had been dried in a water oven for an hour. This, in his opinion, was a most undesirable method, the better way being to dry in vacuum over sulphuric acid. The authors' figures were interesting, but would have been much more so had they stated the amount of nitrogen and phosphoric anhydride they had found. When one extracted with chloroform (as required by the Produce Brokers Contracts) a definite statement should be made as to the method of drying. If the chloroform extract were dried in the oven, there was a long-continued loss of weight.

Mr. G. RUDD THOMPSON said that the proportion of extracted fat with the various solvents was very largely a question of the mode of drying; if an egg were dried in a water oven it would be smelt everywhere, so that something was obviously being lost.

Simple, Useful Forms of Hydrogen Electrode.

BY F. J. CONSIDINE.

(*Read at the Meeting, May* 7, 1924.)

FOR rapid hydrogen ion determinations, especially in the case of biological fluids, the minimal surface contact principle, utilised in the electrodes of Walpole [1913] and of Barendrecht [1915] has much merit.

In large scale process control work, where approximate values suffice, the writer has used modified forms of the above electrodes, and these have proved so convenient and satisfactory that it seems worth while to describe them, as they may perhaps be found of special interest in connection with such inexpensive forms of potentiometer outfit as that well described by Monier-Williams (ANALYST, 1921, XLVI., 315).

Fig. 1 in the accompanying diagram shows a form of electrode for general use. The vessel, made from a broken test tube, is attached to the two-holed rubber stopper through which passes a Walpole platinum wire electrode with mercury drop connection, and also the lower end of the once-bent, stout-walled, capillary bore glass tube. The horizontal portion of this tube acts as a fixed plunger within the sliding glass tube with well lubricated rubber sleeve, to form a simple suction filling device on the Barendrecht principle. The assembled apparatus is supported by a movable clamp grasping the horizontal portion of the capillary tube near the bend. This clamp, by movement upon a retort stand, permits the apparatus to be raised or lowered at will.

To use the electrode, the outer tube of the glass filler is withdrawn to the extremity of the plunger, and, controlled by the Hoffman screw clip, a current of purified hydrogen is passed through the system to expel air and saturate the electrode. The fluid to be tested is then brought into contact with the lower end of the electrode vessel, the gas shut off, and, by appropriate manipulation of the filler, the fluid is drawn up into surface contact with the platinum point, when, after the usual adjustment of salt bridge and wire connections, under favourable conditions a voltage reading may be taken at once.

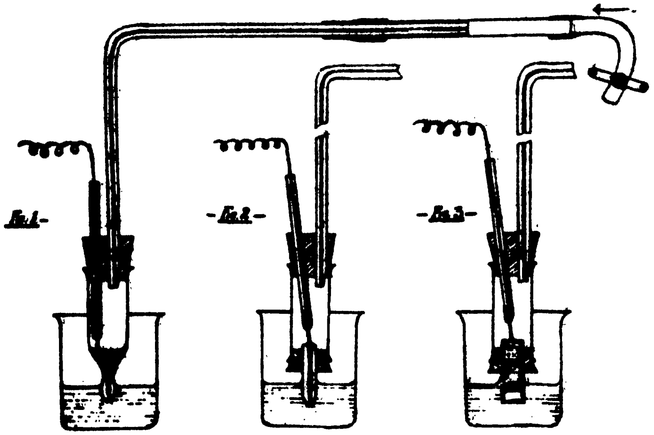

Fig. 2 illustrates a "micro" modification of the foregoing, and is used where very small quantities of a sample are to be tested. Quite satisfactory readings have been obtained with 0·1 c.c. or less of fluid with this electrode. The short, lower capillary tube is of neutral glass, and the stopper hole for the electrode is bored somewhat obliquely to facilitate adjustment of the platinum point directly over the upper orifice of the tube. Walpole's ingenious procedure for testing small quantities of fluid is used here. After thorough flushing of the apparatus with gas a drop or two of the sample fluid is cautiously drawn into the lower end of the short capillary tube, and saturated solution of purified potassium chloride from the vessel below is drawn up after the sample fluid until the latter just makes contact with the platinum point, when a reading can be taken in the usual way. This particular method of exposing the sample fluid to the gas seems to be very effective in securing a rapid equilibrium, as in some cases, where trouble was experienced in securing steady readings with other electrodes, this capillary system gave very rapid and reproducible results. Almost the only objection to this electrode for general use is the need for greater care in its manipulation.

Fig. 3 shows a simple modification of the capillary form and has been found useful for fluids of low conductance. Its use needs no special description.

The simple forms of electrode here described are very easily and cheaply made from materials always at hand, and without expert glass-blowing. They are very strong, the rubber stopper acting as a "shock absorber" in rough handling, and all parts are interchangeable and easily accessible for cleaning.

Some brief working notes in connection with these electrodes may perhaps be of interest.

Palladium chloride is used rather than the platinum salt for coating the platinum wire electrodes. The procedures of Clevenger [1919] for cleaning and coating such electrodes have been found excellent.

For testing the coated electrodes, the potassium hydrogen phthalate solution recommended by Clark [1920] has proved quite satisfactory and much easier to prepare than the buffer mixtures of Walpole, Sörensen, Palitzoch and McIllvaine.

A very simple and good salt bridge used is that described by McClendon [1918], consisting of a thin rubber tube filled with saturated solution of purified potassium chloride, and with ends plugged with pieces of a wooden match, previously boiled in a little of the same solution. This rubber salt bridge connects the sample fluid with a small container of saturated solution of potassium chloride, which also receives a glass siphon provided with a tap kept closed from a home-made Barendrecht [1915] pattern calomel half element. Such calomel half cells, prepared from carefully purified materials and with potassium chloride at full saturation, have proved remarkably stable and are very convenient in use. Protected from light by a covering of suitable paper, they appear to have a very long life. The cylinder hydrogen used is sufficiently purified by passage through Drechsel wash bottles containing alkaline permanganate, alkaline pyrogallol and distilled water. It has been found well to seal glass taps to the intake and outlet tubes of the Drechsel bottles. A "water balance" on the Kipp apparatus principle, in line between gas cylinder and scrubbing train, checks the evil of faulty cylinder valves. A single glass tap, mounted upon the "water balance," is connected by a T-piece with cylinder and scrubbing train, and, in case of a very intractable valve, it is well to disconnect the train while charging up from the cylinder.

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Monier-Williams. ANALYST, 1921, **46**, 315.

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McClendon. *J. Biol. Chem.*, 1918, **33**, 19.

DISCUSSION.

Miss D. JORDAN LLOYD said that she much appreciated the simplicity of design of the apparatus shewn. The first hydrogen electrodes were made upon the simplest mechanical lines, and she regretted the present day tendency to design elaborate apparatus.

Mr. A. Chaston Chapman enquired whether it was necessary to pass the hydrogen for fifteen or twenty minutes in order to obtain a constant reading, as was the case with most of the standard electrodes. The apparatus appeared very much simpler than most of the forms in common use, and he thought that any simplification which could be effected without reducing the sensitiveness of the electrode was very desirable indeed.

Dr. H. E. Cox (who read the paper) said that he had not actually used the apparatus. The connection might be made and under ordinary conditions a voltage reading might be taken almost at once. It was important that the platinum point should only just touch the surface of the liquid and not be immersed to any depth therein.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

OLIVE OIL.

The writer has recently examined two samples of olive oil, each of which had been reported upon as adulterated. The results obtained may be of interest to other analysts.

Sample No. 1. This had an extremely bitter taste, not unlike that of strychnine, and left a burning sensation in the throat.

On analysis it gave the following figures:—Sp. gr., 0·9156; saponification value, 194·4; iodine value, 79·9; free fatty acids, 0·4 per cent.; and $n_D^{40^\circ}$, 1·4613; arachis and cotton-seed oils, absent; Baudouin's test (Furfural and HCl modification), a distinct red, changing to olive-green.

The bitterness and reaction in Baudouin's test seemed the only abnormalities. On referring to Thorpe's Dictionary of Applied Chemistry, Vol. IV., p. 701, I found the following:

> "According to Bourquilot and Vintilesco, olives contain a glucoside, oleoeuropein—a yellow powder with bitter taste—hydrolysable by emulsin, which is present in the fruit, leaves and bark." And under Olive Oil, p. 702: "Olives intended for oil production are gathered just before maturity, as the oil obtained from the barely ripe fruit is much superior in quality to that obtained from fully-ripe or over-ripe fruits."

As it seemed possible that the two bodies might be present in this oil, I shook up some of the oil with water (A) and kept the mixture at a temperature of 80° F., together with some of the oil (B). At the end of ten days the separated oil from sample A was free from bitterness and acridity, and was bland and of a beautiful golden colour.

The analytical figures were very similar to those of the untreated oil, excepting that the free acidity was slightly reduced, and that Baudouin's reaction was almost negative (a very slight and fugitive pink).

Sample B was still bitter and acrid after 14 days in the incubator.

The writer suggests that the abnormal climatic conditions in Spain last year were not favourable to complete hydrolysis taking place before the fruit was pressed (hence the bitterness), and that emulsin and oleoeuropein being present in some stage of hydrolysis, other than the bitter one, may account for the fact that some olive oils give a positive reaction in Baudouin's test.

Sample No. 2. This oil was reported upon as containing 30 to 40 per cent. of arachis oil, this conclusion, presumably, having been arrived at from its behaviour in the British Pharmacopoeia test. The writer has frequently experienced difficulty with this test during the winter months, when the temperature during the 24 hours sometimes falls many degrees below freezing point. As the result of some tests, he would suggest that the B.P. instruction should be altered to read "and kept at a temperature of 15·5° C. for 24 hours."

Some of the oil in question, afterwards reported upon as free from arachis oil (A), some mixed with 15 per cent. of nut oil (B), and some with 10 per cent. of nut oil (C), were subjected to the B.P. test, with this difference, that all three were kept in an incubator at a temperature of 15·5° C. for the required period. The following results were obtained:—

A. Entirely free from crystals; B. A heavy deposit of crystals; C. A very few crystals (so few as to leave one doubtful).

On putting flasks A and C into a water-bath and keeping them at a temperature of 12° C. for an hour, no crystals were deposited in A, but C (with 10 per cent. of nut oil) gave a heavy deposit.

F. F. Shelley.

OSMIUM TETROXIDE AS A REAGENT FOR THE GOLD-BEATERS' SKIN TEST FOR TANNINS.

C. Ainsworth Mitchell (Analyst, 1924, 49, 162) has recently described a quantitative method for the colorimetric estimation of tannins which is based on the use of osmium tetroxide. I have, at the suggestion of Dr. Nierenstein, attempted to use this reagent in connection with the Gold-Beaters' Skin Test for Tannins, as worked out in this laboratory (Atkinson and Hazleton, *Biochem. J.*, 1922, 16, 516; Analyst, 1923, 48, 38; Price, Analyst, 1924, 49, 25), with the result that I have found it to be less delicate than ferrous sulphate or ferrous chloride as recommended by me. Whereas ferrous sulphate is capable of detecting 0·00005 grm. of gallotannin in 1 c.c. of water, osmium tetroxide detects only 0·0001 grm.

Phyllis H. Price.

Biochemical Laboratory,
University of Bristol.

ARSENIC IN PRINTING INK.

During the examination of a sample of proprietary bread it was discovered that the violet ink used for the printing on the wrapper contained appreciable quantities of arsenic. As a result of this discovery a large number of coloured wrappers and labels and a number of coloured papers have been examined to discover whether they contained arsenic. In all, 51 samples have been examined, and of these, nine contained an appreciable quantity of arsenic. These nine samples consisted of two blue papers, five purple papers and two green papers. The quantity of arsenic in one paper faced with a purple colour was about one-third of a grain per square foot.

It was not considered advisable to approach the packers of these goods in order to discover the source of the colours used in the printing. The writer, therefore, made careful inquiries in regard to the composition of printers' inks, and finally, through the courtesy of a large local firm of lithographic printers, he was able to procure nine samples of ink largely used in printing. These consisted of three blues, one violet, one water-violet, one red, two greens and one yellow. All these inks were practically free from arsenic, with the exception of one violet ink, which contained about 10 per cent. of arsenic, calculated as arsenious oxide.

Apart from any danger that might arise from the licking of such brightly coloured paper by young children, it would not seem advisable to allow the possibility of persons obtaining poisonous doses of arsenic without any record that such arsenic had been in their possession.

The presence of the arsenic is due to the use of arsenical lakes in the colours from which the printing ink is made. By the use of these arsenical lakes it is alleged that brighter colours can be obtained, but there seems to be no real reason for their use, especially when one takes into account the possible danger that might arise. This danger is not very urgent, as lakes generally are used on account of their insolubility.

One violet colour used in confectionery was examined, but was found to be free from arsenic. This is merely what was to be expected, as the type of soluble colour used in food is quite different from the lakes used in colour printing.

G. D. Elsdon.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

COUNTY OF LANCASTER.

Annual Report of the County Analyst for 1923.

Of the 4984 samples submitted during the year, 4640 were from the Police, 306 from local authorities, and 2 from other persons. Of the samples submitted by the Police, 365 (7·87 per cent.), and those from local authorities 28 (9·15 per cent.), were adulterated or not up to standard. The total percentage of serious cases, however, was only 2·77 per cent.

Milk.—The number of samples purchased was 3049, and of these, 338 (11·08 per cent.) were adulterated, deficient or dirty. Cow dung exceeding 5 parts per 100,000 was found in 0·62 per cent. of the samples, and in excess of 2 parts, but not of 5 parts, in 2·79 per cent.

In 29 cases milking of the cows was supervised. The highest percentage of fat then found was 5·02, and the average 3·42; whilst the highest and average amounts of solids-not-fat were 9·65 and 8·60 per cent. respectively.

Informal Samples.—The 4301 samples taken without notice included 2456 milks. Of these, 75 milks and 18 other samples were adulterated.

Samples other than Milk.—Five samples of rice contained from 0·1 to 0·4 per cent. of talc. One sample (of 90 examined) of baking powder contained 9 per cent. of calcium sulphate, and one sample of cream of tartar (of 61 examined) contained 28 parts of lead per million.

Ten samples of jam contained salicylic acid in amounts varying from 0·17 to 4 grains per lb. One sample of potted meat, labelled "Free from Preservatives," contained 1·5 grains of boric acid per lb. Official cautions were sent to the manufacturers of potted lobster devoid of lobster, of potted meat containing 30 per cent. extraneous moisture, and of lemon cheese and lemon curd containing 2·9 and 1·9 grains of salicylic acid respectively.

DRUGS.—Four samples of magnesia consisted of magnesium carbonate instead of the oxide. One sample of borax, sold by a grocer, contained 0·5 grain of arsenic per lb.

W. COLLINGWOOD WILLIAMS.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

NOTIFICATION OF WARRANTY.

ON May 15th a firm of druggists was summoned by the Southwark Borough Council at the Lambeth Police Court for selling quinine sulphate which was not of the nature, substance, and quality demanded.

It was explained to the magistrate (Mr. S. Fleming) that the Council's inspector had asked for quinine sulphate and had been supplied with quinine hydrochloride. The defendants pleaded a warranty, but the notice had been sent to the Town Clerk, and not to the purchaser as required by the Sale of Food and Drugs Act, 1889.

For the defence it was urged that the wholesale firm which had supplied the drug had made a mistake, and had sent a more expensive article (quinine hydrochloride) instead of the quinine sulphate ordered. With regard to the legal point, it was submitted that what had been done was a sufficient compliance with the statute.

The Magistrate held that there had not been compliance with the statute, and ordered the defendant to pay 12s. 6d. costs.

There was a second summons against the defendants for selling bicarbonate of soda as purified borax. For the defence it was stated that a mistake had been made. A fine of £5 with £2 16s. 6d. costs was imposed.

RAG-FLOCK ACT CASE.

ON April 29th a firm of upholsterers was summoned by the High Wycombe Borough Council for unlawfully having in their possession flock manufactured from rags intended to be used for upholstery, which flock did not conform to the standard of cleanliness prescribed by the Rag-Flock Regulations, 1912, made by the Local Government Board.

The High Wycombe Town Clerk, for the prosecution, explained that the sample in the present case contained 904 parts of chlorine per 100,000, whereas, under the Rag-Flock Act, only 30 parts per 100,000 was permissible.

Evidence in support of the prosecution was given by Mr. A. P. Davson, F.I.C., to the effect that this was the dirtiest sample of rag-flock he had ever analysed. It consisted of coconut fibre and bagging. He had not discovered any animal fibre, nor had he tested it for grease, but there were particles of foreign dirt. From its appearance he concluded that the material was principally made from gunny bags, with a much smaller amount of coconut fibre. In his opinion, the high proportion of chlorine indicated that the sample was dirty.

A flock manufacturer gave evidence that the sample was composed of coconut fibre and bagging unwashed. Ordinary rag-flock, when washed, lost about 35 per cent. in weight, and there was also the cost of drying. Hence, in his opinion, there was a difference of £10 per ton in the cost of producing unwashed and washed flock.

Another flock manufacturer stated that the sample consisted of about 75 per cent. of bagging and 25 per cent. of fibre. In his opinion the material was unwashed and was unsuitable for upholstery.

The defendants stated that they were under contract for a supply of ordinary rag-flock, but that, owing to a breakdown at the mills, the flock-manufacturers were unable to supply them with their usual material, but said that they could supply a good fibre mixture. The manager of the mills had stated that his firm were prepared to take all risks, and that no guarantee was required, since the fibre mixture did not in any way come under the Rag-Flock Act. Witness produced his invoice showing the small quantities of the fibre mixture which had been purchased, also a letter from the manufacturers confirming the guarantee.

The manager of the mills confirmed the statements of the preceding witness. The material, he said, did not require a guarantee, since it did not come under the Act. He had about 40 customers for this material in Wycombe, but had stopped selling it pending the result of the case.

A consulting chemist called for the defence stated that he had found the sample submitted to him to be a clean fibre mixture, which appeared to consist of coconut, hemp, and jute. He had not detected any woven material, and the sample was also free from wool and cotton. In his opinion the sample did not come under the provisions of the Rag-Flock Act. The high chlorine content was to be expected in view of the fact that coconut fibre was prepared by steeping in salt water. The amount of soluble chlorine was a rough indication of the state of cleanliness of a flock, but had no meaning in the case of a fibre mixture containing coconut fibre.

Counsel for the defence said that if any offence had been committed it was not wilful, and that there had been no intention of evading the Act. He quoted various cases, including one in which Mr. Justice Avory remarked that flock must be manufactured from rag in the ordinary sense of the word.

The Town Clerk, in reply, pointed out that in the case of *Cooper* v. *Swift* it had been laid down that rag-flock was made of woven material, and that even the defence had admitted that there were pieces of woven material in the sample.

The magistrates held that the charge had been proved, and imposed a fine of £7 with £3 costs. Notice of appeal was given.

Meteorological Office, Air Ministry.

ADVISORY COMMITTEE ON ATMOSPHERIC POLLUTION.

REPORT ON OBSERVATIONS IN THE YEAR ENDING MARCH 31ST, 1923.*

THE STANDARD GAUGE.—Thirty-five of these gauges, in which atmospheric impurities accumulate with the rain, were in operation during the year under 16 authorities. The results are expressed, as in the last report, in terms of metric tons per 100 sq. kilometres, or in grms. per 100 sq. metres. Generally speaking, a marked deduction in atmospheric pollution was noticed during the year, and a table is given showing the places with the highest and lowest deposits, including tar, for summer (S) and winter (W), 1922–23:—(S) Newcastle, 242 m.t.; Birmingham, 20 m.t.; (W) Newcastle, 195; Glasgow, 20. *Insoluble ash*: (S) Liverpool, 5400; Rothamsted, 478; (W) Liverpool, 5890; Rothamsted, 231. *Carbonaceous matter*: (S) Blackburn, 2696; Southport, 427; (W) Liverpool, 3933; Rothamsted, 121. *Volatile salts*: (S) Glasgow, 2609; Birmingham, 398; (W) Glasgow, 1519; Birmingham, 262. *Soluble ash*: (S) Glasgow, 3280; Rothamsted, 421; (W) Hull, 2903; Rothamsted, 408. *Total solids*: (S) Rochdale, 16698; Rothamsted, 2298; (W) Liverpool, 13565; Rothamsted, 1140.

THE RECORDS OF THE AUTOMATIC FILTER.—This instrument, which measures, hour by hour, the amount of suspended matter in the air, is illustrated by diagrams and numerous graphs showing the suspended impurities on ordinary week-days, Saturdays and Sundays for various parts of London and Glasgow. An increased number of these filters has been in operation during the year with satisfactory results.

THE JET DUST COUNTER.—This counter, described in the previous year's Report (ANALYST, 1924, 49, 35) has been further tested for efficiency. A double-slot arrangement was found satisfactory when the deposit was too thick for examination in foggy weather with the single slot, and details for mounting and counting the records and illustrations of a portable instrument are given. A table of results obtained in Westminster, Cheam, Bloomsbury, S. Kensington, Kew Gardens, Grimsby, Brighton, Hull, etc., is given, and some of the typical results are discussed in detail. Some very interesting facts are brought to light, such as, for example, the sudden appearance of large numbers of mould-cells in the air in autumn; the occasional presence of crystals of soluble salts in the air, and the occurrence of spherical particles of a glass-like nature, the origin of which is at present uncertain. There is no doubt that the extended use of this instrument is being, and will be, of very great service in elucidating problems of air impurity. For instance, records already suggest that the haze often met with over the South of England may be produced in the manufacturing cities of the Midlands and North; and it is hoped to investigate, with the help of the jet dust counter, such problems as the relation of impurity to strength of wind, of bacterial contents to smoke, and so on.

EFFECT OF ATMOSPHERIC POLLUTION UPON VISIBILITY.—Three methods of observation have been brought to bear upon the problem, viz. the method of the illuminated hollow cube; of the searchlight with contrast photometer; and a combination of the two. Details of results are given, and satisfactory agreement was obtained by the different methods. It is expected that valuable information will accrue from observations on variations of obstruction of light by winter smoke fogs.

D. G. H.

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* M.O. 260. H.M. Stationery Office, Kingsway, W.C.2. Price 4s. 6d. net.

New Methylated-Spirit Regulations.

The Commissioners of Customs and Excise have issued the following notice to makers of methylated spirits regarding the change in the composition of methylated spirits which came in force on May 1:—

1. The Commissioners of Customs and Excise hereby give notice that all mineralised methylated spirits made on and after May 1 must consist of: Plain British spirits or unsweetened foreign spirits or rum (90 per cent.), wood naphtha or methyl alcohol (9½ per cent.), and crude pyridine (½ per cent.), and must contain, in addition to this mixture, three-eighths of one per cent. by volume of approved mineral naphtha or petroleum oil, and not less than 0·025 of an ounce of aniline dye (methyl violet) for each 100 gallons of spirits.

2. Crude pyridine which conforms to the following specification will be regarded as suitable for making mineralised methylated spirits:—

(1) It should consist of the bases derived from coal tar, and should not be more deeply coloured than a solution of two c.c. of decinormal iodine dissolved in one litre of water.

(2) It should mix readily and completely with spirits and should give a clear or only slightly opalescent solution when mixed with twice its volume of water.

(3) Ten c.c. of a 1 per cent. solution in water should produce immediately a distinct crystalline precipitate on vigorous shaking after the addition of five c.c. of an aqueous solution of cadmium chloride containing five grammes of the anhydrous fused salt in 100 c.c., and an abundant separation of crystals within ten minutes.

(4) A white precipitate should be formed when 10 c.c. of a one per cent. solution in water are mixed with five c.c. of Nessler's reagent.

(5) One c.c. of crude pyridine dissolved in ten c.c. of distilled water should require not less than 9·5 c.c. of normal sulphuric acid for neutralisation, using Congo Red paper as indicator.

(6) One hundred c.c. slowly heated under the conditions laid down for benzol for motor fuel by the British Engineering Standards Association (B.S. Specification 2 D. 15) should give a distillate of at least 50 c.c. at a temperature of 140° C. and of 90 c.c. at 160° C.

3. Every maker of mineralised methylated spirits must provide in the naphtha warehouse on his methylating premises a suitable vessel of a minimum capacity of 10 gallons for storing the crude pyridine.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Viscosimetric Study of Wheat Starches. O. S. Rask and C. L. Alsberg. (*Cereal Chem.*, 1924, 1, 7–26.) The physico-chemical differences in the gelatinised pastes of wheat starches have been shown by means of their viscosities; the relation between viscosity and concentration is expressed by the equation log viscosity $= m \times$ concentration $+ \log b$, where m and b are constants. High viscosity is observed to be associated with low loaf-volume and protein content and with a high temperature in the locality of growth, and conversely. Starches from winter wheats have higher viscosity than those of spring wheats. For the determinations, 100 to 125 grms. of the flour were made into dough with tap water, washed free from gluten, and the starch first treated with 1 per cent. salt solution to dissolve globulins, then separated and washed, first with water then with alcohol and ether, with the aid of a centrifuge. The product so obtained was dried to constant weight over calcium chloride under reduced pressure and, when stored, contained 3 to 5 per cent. of moisture. The viscosity of pastes varying from 3 to 6 per cent. concentration was measured at 90° C. by a Stormer viscometer, in which a falling weight rotates a cylinder immersed in the viscous liquid; the resistance is measured and calibrated in terms of centipoises (this is practically equivalent to viscosity compared with water = 1·0). The raw starch suspension was warmed by a small burner, with constant movement of the cylinder in the viscometer cup, and the viscosity observed for every 10° C. rise of temperature. A decrease in viscosity occurs between 93° and 95° C., indicating complete gelatinisation. The temperature was then allowed to fall to 90° C. and a number of determinations were made. At this temperature the viscosity in 5·5 per cent. concentration varies between 50 and 213 centipoises, according to the class of wheat. H. E. C.

Nutritive Properties of Wild Rice. C. Kennedy. (*J. Agric. Res.*, 1924, **27**, 219–224.) Comparison has been made by chemical and biological analysis between wild rice (*Zizania aquatica*) and cultivated rice. The chemical analyses of 4 samples of wild rice are as under:—

No.	Moisture. Per Cent.	Ash. Per Cent.	Protein. Per Cent.	Ether extract. Per Cent.	Fibre. Per Cent.	Starch. Per Cent.	Soluble Carbohydrates as dextrose. Per Cent.
1	7·74	1·09	13·36	0·46	1·39	65·26	2·98
2	7·85	1·38	13·97	0·89	1·41	61·69	3·69
3	8·93	1·17	14·62	0·72	1·94	60·47	2·33
4	7·83	1·25	14·40	0·66	1·29	62·03	2·93

Although these results show a high proportion of proteins in wild rice, both these and the mineral salts present are of a variety unsuitable for dietetic purposes. There is just sufficient vitamin *A* to prevent xerophthalmia, but not enough to promote growth. Wild rice is somewhat superior to the cultivated variety as regards its content of vitamin *B*, but still is not an adequate food. H. E. C.

Preserved Spinach. L. Gobert. (*Ann. Falsificat.*, 1924, **17**, 158–160).—Microscopic examination of a sample of preserved spinach suspected of adulteration with foreign leaves revealed the epidermis and parenchyma of the spinach leaf, round pollen grains, the rounded anthers (from 0·2 to 0·5 mm. in diameter and having a mammillary surface), and vestiges of the staminal threads. Only the male flowers were found, the absence of female flowers being probably explained by the practice, among the seed-growers, of uprooting the male plants after fertilisation. Such plants are doubtless bought at a cheap price, and preserved, and although not adulterated, are of inferior quality, especially if more or less fibrous parts of the petioles should remain in the product. Proper plant should, however, remove such fibrous material. T. H. P.

The Fining of Wine with Potassium Ferrocyanide. H. Bosselmann (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 209–214.) The practice of fining and removing iron from wine by means of ferrocyanide, as recently described in an official pamphlet in Germany, may lead to the formation of small quantities of hydrocyanic acid. For the detection or estimation of hydrocyanic acid in wine the usual silver nitrate test is not satisfactory, either when applied directly or after distillation with dilute acid. The following process is recommended:—About 300 c.c. of the wine are heated to boiling in a round-bottomed flask connected with a ball reflux condenser. Through the boiling liquid is passed a stream of carbon dioxide which is led from the top of the condenser through small wash bottles containing (*a*) 30 c.c. of 6 per cent. sodium bicarbonate solution, (*b*) 10 c.c. of 0·01 *N* mercuric chloride solution acidified with 1 c.c. of dilute hydrochloric acid, and through 2 test tubes, each containing 10 c.c. of 0·01 *N* silver nitrate solution. The bottles (*a*) and (*b*) are heated in hot water baths to 90° C. and 50–60° C. respectively. When all the hydrocyanic acid is carried over, as indicated by testing with a further quantity of silver nitrate, the solution is back-titrated with ammonium thiocyanate, as in Volhard's method. The silver cyanide precipitate may be identified by the formation of Prussian blue in the usual way. Details are given of the experimental fining of certain wines. Free hydrocyanic acid is only found when an excessive quantity of ferrocyanide is added, and is apparently formed by the action of the organic acids present in the wine. When testing for hydrocyanic acid it is important first to remove any precipitate of Prussian blue which may be present; otherwise it will decompose at the boiling point.

H. E. C.

Fatty Acids of Camel Hump Fat. N. McClelland. (*J. Soc. Chem. Ind.*, 1924, **43**, 164 T.)—Values for the fat obtained from the hump of a fully-grown young camel (total 80 lbs.) were as follows:—Sp. gr. at 100/15° C., 0·872; m.pt., 35–36° C.; insoluble fatty acids, 92 per cent.; unsaponifiable matter, 0·4 per cent.; saponification value, 205–206; iodine value, 37·4; m.pt. of fatty acids, 45–46° C.; neutralisation value of solid fatty acids, 210–211. *Solid acids.*—The first main fraction (b.pt. 195–200° C.) was identified as ethyl palmitate by its melting pt., melting pt. of the methyl ester, and melting pt. and molecular weight of the free acid. The second main fraction (b.pt. 210–215° C.) was similarly identified as ethyl stearate. The residue (boiling above 215° C.) was found to consist of impure stearic acid. *Liquid acids.*—Acids yielding tetrabromides and hexabromides were not present. The liquid acid was converted, through the potassium and lead salts, into the acid, and this into the barium salt, which was recrystallised, and the acid liberated. The oily acid melted at 11° C., and had a molecular weight of 280, and was proved to be oleic acid. The approximate composition of the mixed fatty acids was found to be: Palmitic, 37; stearic, 16; and oleic, 47 per cent.

D. G. H.

Bellier's Reaction applied to the Detection of Adulteration of Cacao Butter. N. B. Clemencet. (*Ann. Falsificat.*, 1924, **17**, 146–153.)—When present to the extent of 10 per cent. in admixture with colourless, neutral vaseline oil, pure cacao butter should give a positive violet reaction with Bellier's reagent (1·5 grms. resorcinol in 1 litre of benzene), stable for about 10 minutes, but changing gradually into pink. This reaction may, therefore, be used as a criterion of the purity of cacao butter, and, by determining the minimum quantity of a mixture capable of showing the reaction, as a means of estimating approximately the proportion of cacao butter present in such mixture. T. H. P.

The Kreis Test. G. E. Holm and G. R. Greenbank. (*Ind. Eng. Chem.*, **1924, 16,** 518.)—Linolic, linolenic, and ricinoleic acids give a reaction with the Kreis reagent when they have absorbed oxygen, but the proportion of colour produced is less than that obtained with oleic acid. As regards ricinoleic acid, the relative proportion of colour it will give is small, and the rate of absorption of oxygen is slow. The odours produced in the different oxidations show that oleic acid is the main constituent of fats and oils that is concerned in the production of tallow flavour or rancidity (*cf.* ANALYST, 1923, **48,** 608). W. P. S.

Nature of the Oil of Soya Bean Miso. R. Kodama. (*Ind. Eng. Chem.*, **1924, 16,** 523.)—A fermented soya bean preparation, known as "miso," is one of the staple food products of Japan. It contains from 4·8 to 11·5 per cent. of liquid or semi-solid oil having the following characteristics:—Sp. gr., 0·9466 to 0·9471; acid value, 22·49 to 77·69; saponification value, 211·0 to 235·3; iodine value, 93·5 to 97·96; Hehner value, 93·53 to 97·96; Reichert-Meissl value, 3·56 to 7·46. When decolorised with charcoal, the oil becomes nearly solid, the iodine value is decreased by 30 to 40 units, the saponification value increased by 4 to 60 units, but the acid value is unaffected. W. P. S.

Analysis of Gelatin Ash. E. Cattelain. (*J. Pharm. Chim.*, 1924, **29,** 414–417.)—The composition of the mineral matter obtained from a representative commercial gelatin manufactured for pharmaceutical and bacteriological purposes, was found to be as follows:—*Soluble*: Lime, 53·36; magnesia, trace; ferric oxide, 1·22; phosphoric anhydride, 5·40; and sulphuric anhydride, 36·36 per cent. *Insoluble*: Silica, 3·36 per cent. The average composition of the ash of the purest commercial gelatins shows the sum of the lime and sulphuric anhydride to be 90 per cent., and that of the silica, iron sesquioxide and phosphoric anhydride to be 10 per cent. Traces of copper are very frequently found, whilst manganese is almost constantly present, and may, to a certain extent, influence bacteriological cultures. It may be noted that the copper was estimated colorimetrically by dissolving 1 grm. of the ash in 10 c.c. of 25 per cent. sulphuric acid, making up the solution to 20 c.c. with ammonium hydroxide solution, and comparing the colour with that shown by a solution of copper sulphate containing 1 mgrm. of copper per c.c. Manganese was also estimated colorimetrically by Bertrand's method of comparison with a standard solution of crystalline manganous sulphate, acidified with nitric acid, the ash being dissolved in 25 per cent. nitric acid, and the solutions in each case treated with 5 drops of 10 per cent. silver nitrate solution, and 0·25 grm. of potassium persulphate. The maximum amount of manganese found was 0·10 per cent. D. G. H.

Estimation of Eugenol in Clove Oil. J. A. L. Bouma. (*Pharm. Weekblad*, 1924, **61**, 249–250.)—It is shown that the factor for converting the reduced silver into eugenol in van Eck's method of estimation (ANALYST, 1923, **48**, 567) is not a constant, but varies with the amount of eugenol present and the duration of heating. Thus, by varying the conditions, factors ranging from 1·17 to 1·83 were obtained, and the conclusion is therefore drawn that the reaction is unsuitable for the quantitative estimation of eugenol.

Estimation of Santonin in Wormseed. O. P. A. Schaap. (*Pharm. Weekblad*, 1924, **61**, 277–280.)—Five grms. of the drug are dried in a desiccator and mixed with 1·5 grms. of slaked lime and sufficient water to form a paste. After standing 24 hours the mass is treated with 100 c.c. of water, and boiled for 30 minutes, after which a solution of 5·5 grms. of zinc sulphate in a little water is added, and the boiling continued for 10 minutes. The liquid is filtered, the filter and residue boiled for 15 minutes with 100 c.c. of water, and this new extract filtered. The united filtrates containing the santonin, probably in the form of zinc santoninate, are treated with 3 c.c. of 30 per cent. nitric acid and evaporated to dryness, and the residue treated with 25 c.c. of water and again evaporated. Ten c.c. of water and 2 grms. of calcium carbonate and a little sand are then added, and the mixture cooled, transferred to a separator, and shaken for 2 minutes with 50 c.c. of chloroform. The extract is filtered and 40 c.c. of the filtrate (=4 grms. of the sample) are evaporated, the residue dissolved in 5 c.c. of methyl alcohol, and the solution diluted with 35 c.c. of water at about 60° C. and left for 24 hours. The crystals of santonin are then collected on a weighed filter, washed with five successive quantities of 2 c.c. of water, and dried at 100° C. By this method practically the whole of the resin acids in the drug is eliminated.

Biochemical, Bacteriological, etc.

Alkyl-chloro-malonamides. The Influence of Homology on Taste. A. W. Dox and B. Houston. (*J. Amer. Chem. Soc.*, 1924, **46**, 1278–1281.)—All the normal alkyl-chloro-malonides have a decidedly sweet taste, the *n*-butyl derivative being both bitter and sweet. Dichloromalonamide, and ethyl-, *n*-propyl-, *iso*-propyl-, and *n*-butylchloromalonamides have about nine times the sweetening power of sucrose. The maximum sweetness was observed with the *n*-hexyl derivative, of which 1 part in 5000 of water had the sweetness of a 6 per cent. solution of sucrose. This observation was confirmed by comparison with a solution of "dulcin" (*p*-phenetyl urea), which is stated to be 250 times as sweet as sucrose. On the other hand, the *iso*-butyl, *iso*-amyl and benzyl derivatives stand in sharp contrast to the other members of the series, being decidedly bitter and without any sweet taste. No constant variation in sweetness could be established in the series. The sweet taste of the hexyl derivative is free from any disagreeable after-taste, but the compound has the drawback of being difficult to dissolve in water.

Rubidium and Caesium Creatinine Picrates. I. Greenwald and J. Gross. (*J. Biol. Chem.*, 1924, **59**, 613–614.)—Since Jaffé's creatinine potassium picrate, in spite of its slight solubility, is too soluble for use in isolating very small amounts of creatinine from large quantities of biological material, an attempt was made to prepare other double picrates of creatinine. The hot solutions of creatinine, picric acid and the sulphate or chloride of the metal or radicle were mixed in equivalent quantities, and a little sodium acetate was added. The crystals obtained on cooling were recrystallised from hot water. With lithium and sodium only creatinine picrate was obtained, and with ammonium picrate and trimethyl ammonium picrate mixtures of these with creatinine picrate were obtained. After several recrystallisations, however, pure creatinine picrate was isolated. Definite double salts were obtained with rubidium and caesium, viz. creatinine rubidium picrate and creatinine caesium picrate. Results of analyses are given. Both salts are considerably less soluble than creatinine potassium picrate, and it is proposed to use the rubidium salt, which is the least soluble of the three, in an attempt to isolate creatinine from blood. P. H. P.

Chemistry of Jaffé's Reaction for Creatinine. I. Greenwald and J. Gross. (*J. Biol. Chem.*, 1924, **59**, 601–612.)—Little is known of the chemical changes involved in the reaction of Jaffé (*Z. physiol. Chem.*, 1886, **10**, 391) for creatinine. From experiments to determine the rate of destruction of picric acid in this reaction, which are described and results of which are tabulated, it was found that only 1 mol. of picric acid appears to be required for each mol. of creatinine, although a considerable excess (2·5 mols.) is necessary to complete the reaction. Both picric acid and creatinine may be quantitatively recovered if the alkaline mixture, made as recommended by Folin (*Z. physiol. Chem.*, 1904, **41**, 223) for a creatinine estimation, is re-acidified within 10 minutes, but after 30 minutes the former can no longer be quantitatively recovered, since the colour of the alkaline mixture begins to fade. A red precipitate was obtained on adding hydrochloric acid to a concentrated mixture of creatinine, excess of sodium picrate and slight excess of sodium hydroxide. After washing and drying this formed a brilliant red powder which is, apparently, a tautomeric form of creatinine picrate, since it became creatinine picrate at 139° C. The formation of this substance appears to be responsible for the red colour in Jaffé's reaction for creatinine, which Chapman (ANALYST, 1909, **34**, 475) thought was due to the formation of the sodium salts of picramic acid (monoamino-dinitrophenol) and diamino-nitrophenol. Certain other substances, similar in constitution to picric acid, did not give the reaction. A slight intensification of the colours of alkaline solutions of the salts of trinitrobenzene, trinitrotoluene and trinitrobenzoic acid was observed.

P. H. P.

Estimation of Histamine. M. T. Hanke and K. K. Koessler. (*J. Biol. Chem.*, 1924, **59**, 879–888.)—The work reported in Papers XVII. and XVIII. of this series (*J. Biol. Chem.*, 1924, **59**, 835 and 855) brings definite proof that the normal bacterial inhabitants of the alimentary tract have the faculty of converting the innocuous amino acids, histidine and tyrosine, into the highly toxic amines, histamine and tyramine. A purely chemical method is described for the estimation of histamine in the liver, intestinal contents, and faeces of animals. The material to be examined for histamine is hydrolysed by heating with 37 per cent. hydrochloric acid for 30 hours, and the black insoluble residue formed is filtered off. The acid is removed by distillation *in vacuo* at 60° C. from the same flask. The residue is then dissolved in water, and the mixture is treated with excess of lime and a volume of alcohol equal to one-half the volume of water added. The mixture is distilled *in vacuo*, whereby the ammonia is removed, and filtered to remove the humin. The colouring matter of the alkaline solution is progressively lost during the subsequent steps of the process, but, if not completely removed, mercuric sulphide precipitated in the solution at the end adsorbs it. The alkaline filtrate is acidified with hydrochloric acid, concentrated on a water-bath, and treated with phosphotungstic acid in acid solution. The phosphotungstates are removed by filtration, and the washed precipitate, containing the histamine, is macerated in a mortar with a hot, concentrated barium hydroxide solution, heated on a water-bath for 1 hour, filtered and the barium removed from the filtrate with sulphuric acid. The acid filtrate is concentrated on the water-bath, the residue diluted with water, made alkaline, and extracted exhaustively with amyl alcohol, which removes histamine quantitatively. The alcohol extracts are extracted five times with *N* sulphuric acid; the histamine passes into the aqueous acid layer, which is neutralised with barium hydroxide, filtered and concentrated; the residue is dissolved in water, and the solution made alkaline and extracted with amyl alcohol as above. This is done three times in all, and is very important. The final residue is dissolved in water and estimated colorimetrically (*J. Biol. Chem.*, **1919**, **39**, 597). Human faeces (500 to 600 grms. from normal individuals) have yielded from 6 to 20 mgrms. of histamine. The cæcal content (600 and 1200 c.c.) contained 2 and 7 mgrms. of histamine respectively. One human liver examined yielded none. Dog faeces (150 grms.) contained 5·3 mgrms., and the liver of the same dog yielded 6 mgrms. of histamine. The intestinal contents, intestinal tract, and liver of two normal guinea pigs did not yield histamine. P. H. P.

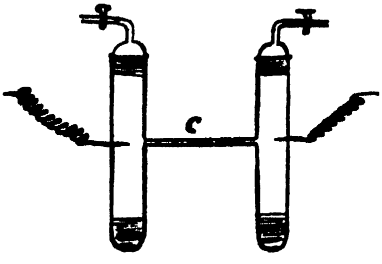
Use of Guaiacol for Measuring the activity of Oxidase Preparations. P. Fleury. (*J. Pharm. Chim.*, 1924, **29**, 402–414.)—Under certain conditions guaiacoquinone, a product of oxidation of guaiacol, can be estimated colorimetrically, the oxidation of guaiacol in the presence of air and laccase being very nearly complete, but the yield is slightly variable, possibly owing to the decoloration of guaiacoquinone solutions by excess of guaiacol. The laccase was found to be practically unaltered at the end of the reaction. Since, however, the colouring matter tends to precipitate and cannot be redissolved in excess of water, owing to the formation of a colloidal solution, it must be separated before any estimation can be made ; for this purpose chloroform is used, as it at once removes practically the whole of the guaiacoquinone and renders the reaction more stable. It was found that a solution of 46·5 mgrs. of guaiacoquinone in a litre of chloroform had the same tint as a $N/100$ solution of iodine observed through a thickness of 10 mm. The chloroform solution must be prepared by directly extracting the water solution of guaiacol treated with laccase, and attention must be paid to the relation between the proportion of guaiacoquinone and the time of contact of the colloidal solution of guaiacoquinone with the guaiacol. For details of working, the original paper should be consulted. D. G. H.

Influence of the Bactericidal Power of Raw Milk on Lactic Acid Organisms in Sterilised Milk. P. Mazé. (*Comptes rend.*, 1924, **178**, 1434–1436.) —Occasionally in the spring, and less frequently in the autumn, lactic acid organisms which have been kept in sterile milk lose their power of acidifying pasteurised skim milk or cream. Apparently the bactericidal power of the fresh milk undergoes reinforcement owing to the change of the cows' diet. T. H. P.

Toxicological and Forensic.

A Case of Mercuric Chloride Poisoning. Limit of Sensitiveness of the test for Mercury in Toxicology. A. Sartori. (*Chem. Zeit.*, 1924, **48**, 141–142.) —An unusual and fatal case of corrosive sublimate poisoning is described which was due to the use of a very weak solution of this salt as a vaginal wash. Subsequent examination of parts of the body by the usual method of the destruction of organic matter by potassium chlorate and hydrochloric acid, followed by hydrogen sulphide precipitation, yielded negative results, even when working on quite large quantities of the material. Similar tests on the intestines and kidneys showed only a trace of mercury. Experiments made show that the most delicate test is the deposition of the mercury on copper foil and the preparation therefrom of mercuric iodide in the manner recommended by Jannasch (*Zeitsch. anorg. Chem.*, **12**, 43). This will detect 0·05 mgrm. in aqueous solution, or about 2 mgrms. in admixture with organic matter. H. E. C.

Spectroscopic Identification of Mercury. Naoum. (*Chem. Zeit.*, 1924, **48**, 311).—Quantities of mercury too small for detection by chemical methods may be identified spectroscopically. The substance in form of a dry powder is placed in the two limbs of the tube figured, which is then exhausted at the pump. The limbs are heated in a water-bath and, on passing a current through the vacuum tube thus produced, the mercury spectrum may be identified in the cross piece (c). Still greater delicacy is obtained by subliming the mercury on to pure gold-leaf, which readily absorbs it; the gold-leaf is then examined spectroscopically in the above manner.



H. E. C.

Organic Analysis.

Oxalic Acid as a Standard Substance for Titrations. W. D. Treadwell and H. Johner. (*Helv. Chim. Acta*, 1924, **7**, 528–534.)—Anhydrous oxalic acid is difficult to obtain and is too hygroscopic for general use; the ordinary hydrated acid contains occluded water, which makes it useless as a standard substance. The vapour pressure curve has been investigated by the dew-point method and the equilibrium is worked out in detail. For purification, the acid is recrystallised from hydrochloric acid of sp. gr. 1·07, as directed by Winkler, but the method of drying given by Lunge (6 hours at 70–80° C.) is not satisfactory. The anhydrous acid can be obtained pure by the method first given by Hampe (*Chem. Zeit.*, 1883, **7**, 73, 106), which consists in drying the crystals at 100° C., then subliming them *in vacuo* at 140° C., again drying them at 100° C., and finally cooling them in a desiccator over calcium chloride. For analytical purposes the hydrated acid is best; it may be prepared by passing air first through a large wash bottle containing equal parts of hydrated and of anhydrous acid, and then through a small weighed tube containing the powdered crystals of the hydrated acid until this is entirely free from occluded water, as is shown by the weight becoming constant. H. E. C.

Estimation of Organic Phosphorus. E. J. Baumann. (*J. Biol. Chem.*, 1924, **59**, 667–674.)—Experiments described shew that serious losses of phosphorus may occur in the wet ashing used in the micro-methods for estimating phosphorus, these being due to (*a*) Volatilisation of phosphoric acid; and (*b*) conversion of orthophosphoric to meta- and pyrophosphoric acids. A process has been developed to avoid these losses, and it allows from 0·02 mgrm. to several mgrms. of phosphorus to be estimated accurately and easily. By heating with 20 to 30 per cent. hydrogen peroxide and sulphuric acid, organic matter oxidisable with difficulty can be oxidised in a short time, and at a comparatively low temperature, sufficiently to liberate all combined phosphorus as orthophosphoric acid. Free saturated acids formed from fats do not interfere in the estimation. Applications of the method are described for estimations of lipoid phosphorus in animal tissues and fluids by using the Bloor alcohol and ether extraction process, oxidising with hydrogen peroxide and sulphuric acid, and finally estimating the phosphorus by the colorimetric method of Bell and Doisy (*J. Biol. Chem.*, 1920, **54**, 55). The tissue is ground to a paste, a small sample weighed out, mixed with plaster of Paris and dried over sulphuric acid in a vacuum desiccator. Then it is thoroughly pulverised in a mortar, with the aid of washed powdered glass, and transferred to a Gooch crucible on an asbestos mat. The substance is next extracted in a Wiley extraction apparatus with anhydrous ether during the day (6 hours), and with anhydrous alcohol overnight. This process is repeated during the next 24 hours, and, finally, the substance is extracted a third day with alcohol. In this 3 day-extraction, 95 per cent. of the material soluble in alcohol-ether which can be extracted in 10 days is obtained. It is concentrated to a few c.c. and dried overnight in the vacuum dessicator. The fatty material is dissolved in anhydrous chloroform, filtered several times (in case plaster of Paris is still present), evaporated nearly to dryness, dried in a vacuum dessicator over sulphuric acid for 15 hours and weighed, then re-dissolved in a definite amount of chloroform ready for the estimation. All the apparatus used is washed with the various solvents or extracted. The greatest source of error lies in the extraction processes. The same process as for lipoid phosphorus estimations of blood, full details of which are given, is then employed, but usually more hydrogen peroxide is needed for the oxidation. P .H. P.

Testing Decolorising Carbons. J. E. Temple and P. Mahler. (*Ind. Eng. Chem.*, 1924, **16**, 498–500.)—With a Hess-Ives tint photometer and a given solution, the action of any carbon on that solution may be represented on logarithmic paper by a straight line, and the comparative value of different carbons acting on the same solution may be easily deduced. It is usually sufficient to follow the decolorisation only with the light which is most strongly absorbed; in the case of many commercial liquids this is the blue-violet, since they are usually coloured red, yellow or brown and absorb but little red or yellow light. Of course, when decolorising a blue-violet solution, absorption of red or green light would be measured. The action of carbons on one solution cannot be applied to another solution; tests made on iodine solution or by other so-called standard methods were of little practical value, and the method of simply recording the decolorising power on any given solution as 70 or 90 is misleading. The adsorption of colour by carbon is an equilibrium reaction, but substances other than colouring matters assist in determining the equilibrium values. W. P. S.

Estimation of Fat, Wax and Resin in Cotton. P. H. Clifford, L. Higginbotham and R. G. Fargher. (*J. Textile Inst.*, 1924, **15**, T 120.)—Solvents extract from cotton, fat, fatty acids, wax-alcohols, acids and esters, phytosterol, phytosterol glucoside, hydrocarbons, resin acids and resenes, so that it is impossible to eliminate one group and leave the others unaffected. Experiments have been carried out for varying periods of time with the use of different solvents, such as chloroform, benzene, carbon tetrachloride, ether, petroleum spirit, etc. The Soxhlet apparatus is made of tinned copper large enough for 100 grms. of cotton and 1200 c.c. of solvent, and is of two forms:—(1) For extracting the material at the ordinary temperature; (2) for extracting the material at the boiling point of the solvent. The drying of the extract is recommended to be carried out at 15 mm. pressure and 80° C., rather than at 760 mm. and at 100 to 110° C., as no darkening occurs, and the method is quicker. To obtain uniform results rigid conditions must be maintained. For fat, wax and resin, chloroform should be used in the hot Soxhlet for 6 hours. For fat and wax only, carbon tetrachloride should be used in an ordinary Soxhlet extractor. The presence of size does not materially interfere with the removal of the natural wax and resin. For large-scale extractions, chlorinated solvents should be avoided, since they are susceptible to hydrolysis, with liberation of free hydrochloric acid which might tender the cotton when steaming off the last traces of solvent after extraction.

R. F. I.

Chrome-leather Analysis. D. Woodroffe. (*J. Soc. Leather Trades Chem.*, 1924, 8, 194.)—In the estimation of moisture, a recommendation is made to use air-tight aluminium boxes, 3½ ins. diam. × ½ in. deep, in which the leather can be weighed out, dried, transferred to the desiccator and re-weighed, all in the same vessel. These are preferable to weighing-bottles. On the question of fat-estimation the author has done some work on the use of different solvents in succession, and also on the effect of drying the leather before extraction. He recommends that the leather be dried for 2 to 3 hours at 100–110° C. and extracted with petroleum spirit for 3 hours. The solvent (b.pt. 40–60° C.) should siphon over at least twelve times. The extract is evaporated and dried in a water-oven for 2 hours, or to constant weight. *Basicity of the leather*: The method for this estimation as carried out by Meunier and Chambard is compared with the older method, in which the SO_4-radicle is estimated by treatment of the dried ether-extracted and water-extracted leather with fuming nitric acid in the cold for three days. Experiments are described which tend to show that Meunier and Chambard's method is only satisfactory for freshly-tanned wet leather, and not for finished dried leather. Results are given in terms of the new official form of expression (per cent. chromium combined with hydroxyl), and also the older English method (grms. of SO_4 per 52 grms. of chromium). A method for estimating alumina is quoted, in which aluminium and chromium are estimated together gravimetrically, and the aluminium calculated after subtracting the chromium as estimated volumetrically. Results are said to be too high, owing to salts being precipitated with the hydroxides. R. F. I.

Standard Methods for the Analysis of Rubber Articles. (*Ind. Eng. Chem.*, 1924, 16, 397–402.)—The following methods of analysis are recommended by the Committee on Methods of Analysis of the Division of Rubber Chemistry of the American Chemical Society. *Acetone Extract.*—Two grms. of the sample, ground to pass a No. 14 sieve or cut into small pieces, are extracted in a siphon apparatus for four hours, or for twelve hours in the case of hard rubber; the acetone solution is then evaporated, the residue dried at 70° C., and weighed. From vulcanised material the acetone removes rubber resins, free sulphur, mineral oils and waxes, and a part of any bituminous substances of vulcanised oils that may have been used. The free sulphur is estimated by oxidation with bromine and its quantity deducted from the total extract; the corrected extract thus obtained should not exceed 5 per cent. of the rubber present. *Chloroform extract.*—The sample of rubber, without removal of adhering acetone, is extracted for four hours with chloroform, the chloroform solution is evaporated, and the residue dried at 70° C., and weighed; this residue consists of a portion of the bituminous substances and serves as an indication of their presence. *Alcoholic alkali extract.*—The rubber after the two previous extractions is dried and boiled with 50 c.c. of N alcoholic sodium hydroxide solution for four hours under a reflux condenser, the solution is then filtered, the rubber washed with hot alcohol and then with water, the filtrate is evaporated to dryness, the residue mixed with water, acidified with hydrochloric acid and extracted with ether; the residue obtained on evaporating the ethereal solution is dried and weighed; it represents the amount of rubber substitutes present. *Total sulphur.*—This is estimated by treating 0·5 grm. of the rubber with 15 c.c. of nitric acid saturated with bromine for one hour, heating the mixture on a water-bath one hour and evaporating it to dryness; the residue is mixed with 3 c.c. of nitric acid and 5 grms. of sodium carbonate, dried, fused, and the sulphate estimated by precipitation as barium sulphate. An alternative method is given for the estimation of total sulphur other than that present as barium sulphate, if this has been used as a filler; the sample is treated with a mixture of zinc oxide and nitric acid (prepared by adding 200 grms. of zinc oxide to 1 litre of nitric acid), oxidised with fuming nitric acid and bromine, the mixture evaporated to dryness, the residue baked, dissolved in hydrochloric acid, the solution filtered and the sulphate estimated in the filtrate. *Ash.*—The ash of the sample should be estimated; it represents the non-volatile fillers together with their reaction products with sulphur. *Rubber.*—This is estimated indirectly after allowance has been made for carbonates, cellulose, and hydrocarbons of mineral rubbers. Special methods are also given for the analysis of materials which contain glue, carbon, antimony and waxy hydrocarbons. W. P. S.

Inorganic Analysis.

Precipitation of Zinc as Sulphide. O. Hackl. (*Chem. Zeit.*, 1924, **48**, 326.)—In Schneider's method for the precipitation of zinc sulphide from feebly acid (sulphuric) solutions, proper neutralisation is a decisive factor for the success of the operation. The solution, to which a piece of Congo paper is added as indicator, should not be too dilute; it is made distinctly ammoniacal, acidified with sulphuric acid, then treated with strong ammonia solution, drop by drop, until the alkaline reaction persists after 5 to 10 seconds' stirring. The prescribed small excess of sulphuric acid, calculated on the subsequent dilution desired, is then added. W. R. S.

Volumetric Assay of Iron Ores by means of Titanous Chloride. L. Brandt. (*Chem. Zeit.*, 1924, **48**, 265–266, 270–271.)—The titanous chloride method of Knecht and Hibbert possesses the advantage over the usual volumetric methods of not requiring preliminary reduction of the iron to the ferrous state. It was tested as to its applicability to the assay of iron ores, the tests bearing on the uniformity and proportionality of the results and the question of the interference of other elements. Thiocyanate was used as internal indicator, being added either at the outset or after the bulk of the ferric salt had been reduced. The titrations were carried out at ordinary temperatures; it was found unnecessary to maintain an atmosphere of inert gas over the solution undergoing titration, but the stock solution of titanous chloride must be stored in a bottle communicating with a source of carbon dioxide; in this manner it kept unchanged for several weeks. The process was found to be simple and accurate, and to give almost exactly proportional results. The only interfering metals are copper, antimony, vanadium, and platinum, whilst larger quantities of chromium and cobalt affect the end-point more or less by yielding coloured solutions. The interference of platinum (derived from the crucible in the case of a bisulphate fusion) is smaller than in the permanganate titration. Antimony, cobalt, and vanadium occur very rarely and in negligible quantities; for practical purposes, copper is the only metal which seriously interferes, being quantitatively reduced to the cuprous state.

W. R. S.

Detection of Cobalt and Nickel. C. C. Palit. (*Chem. News*, 1924, **128**, 293–294.)—A few drops of the solution are added to a saturated solution of sodium bicarbonate. The cold liquid is shaken with a little bromine water; an apple-green colour indicates presence of cobalt. The solution is then heated, when a bluish-black precipitate (forming a filmy deposit on the glass) shows the presence of nickel. W. R. S.

Analysis of Sodium Peroxide. E. Bosshard and E. Furrer. (*Helv. Chim. Acta*, 1924, **7**, 486–489.)—Two methods are available for the estimation of the active oxygen in sodium peroxide, *viz.* gasometric and volumetric. For the former 0·2 to 0·6 grm. of the powder, weighed in a closed tube, is introduced into a small flask fitted with a fine dropping funnel and connected with a Bunte burette; 15 c.c. of 1:10 sulphuric acid and 3 drops of a saturated solution of cobalt nitrate are slowly added from the dropping funnel, and afterwards the flask is gently warmed. The gas evolved may be measured over brine or potassium hydroxide solution, the latter having the advantage of absorbing any carbon dioxide. The difficulty with the permanganate titration method is the decomposition brought about by the heat of hydration of the sodium peroxide; this may be overcome by the following method, which gives results identical with those of the gasometric method:—From 0·2 to 0·4 grm. of the peroxide is mixed in a mortar with 3 to 5 grms. of powdered boric acid. Then 100 c.c. of water and 10 c.c. of 1:5 sulphuric acid are added, and the resultant hydrogen peroxide is titrated in the usual manner with 0·1 *N* potassium permanganate solution. H. E. C.

Detection of Phosphoric Acid. F. Frey. (*Chem. Zeit.*, 1924, **48**, 281.)—Feigl's benzidine reaction (ANALYST, 1923, **47**, 93) was found to be easily carried out and extremely sensitive, as it indicated less than 0·000,001 grm. of P_2O_5 in 0·1 c.c. A small quantity of the solution is acidified with nitric acid and treated with twice its volume of the usual molybdate reagent. The solution is warmed for a short time, and run through a filter paper of close texture. A drop of ammonia solution is brought into the apex of the filter by means of a glass rod, and a drop of benzidine acetate solution poured upon the paper, when the intense blue coloration is produced if phosphoric acid is present. The reaction has the additional advantage that the yellow precipitate of molybdic acid, which sometimes forms, cannot be mistaken for ammonium phosphomolybdate. W. R. S.

Physical Methods, Apparatus, etc.

Simple Method for determining the Approximate Index of Refraction of Liquids with a Common Microscope. C. C. Kiplinger. (*J. Chem. Soc.*, **1924, 125,** 963–965.)—A Johannsen auxiliary lens may act as a telescopic objective, the optical system of the microscope becoming the eyepiece. If such a spherical lens is placed above, and in contact with, a glass slide on the stage, the lens being brought under the objective and the whole system focussed on an object one or two feet from the microscope, the introduction of a drop of liquid between the lens and the slide changes the focus of the optical system by an amount proportional to the index of refraction of the liquid.

The auxiliary lens is made by drawing out a soft glass rod to a diameter of about 0·3 mm. and then fusing a bead on the end of a 4 cm. length of the fine rod, any bead failing to give a sharp image being discarded. A bead 1·81 mm. in diameter gave satisfactory results with a 7·5 eyepiece and a 32 mm. objective. If the stem of the bead lens is bent at right angles a few mm. from the spherical tip, the same side of the lens may always be brought next to the glass slide by using the bent stem as an index; in this way the accuracy of the determinations is greatly increased. The focal adjustment is made by moving the graduated draw-tube containing the eye-piece. The sodium lamp of Fales and Morrell (ANALYST, **1922, 47,** 40), improved by replacing the crucible by an alundum cone, is placed 30 to 40 cm. from the microscope, and between the latter and the lamp (to serve as "distant object") is a capillary glass tube, supported vertically on a large cork as base. This object is focussed and the reading on the scale taken. A drop of water is then placed between the slide and the lens, care being taken to keep the water from the upper half of the sphere; the system is then re-focussed by moving the draw-tube only, this being repeated and the mean of the several scale readings taken. The water is then replaced by glycerol and similar readings are taken. From the known refractive indices for water and glycerol, and the reading given by an unknown liquid, the index of the latter may be calculated by interpolation. Results accurate to 0·02 are obtainable in this way. T. H. P.

Receiving Apparatus for Distillation at Low Pressures. W. F. Seyer. (*J. Amer. Chem. Soc.*, 1924, **46,** 1209–1210.)—This apparatus enables a continuous fractional distillation to be made at low pressures without risk of leakage. The tube from the condenser passes through the wall of a large vertical tube having the form of a Liebig condenser, and delivers into a funnel held in position within the large tube by two metal plates supported by constrictions in the wall of the tube. The end of the stem of the funnel is inclined at an angle, so that it can be brought over any one of the openings of a series of tubes sealed into the apparatus, and, in turn, sealed to flasks the sizes of which depend upon the amounts of the various fractions. Clamped to the funnel are two small pieces of iron rod held together at the ends by brass strips, and the funnel, with the fixtures, turns on a glass bearing which rests on a small indentation, so that it swings about easily when a magnet is brought near to either of the iron pieces. The whole apparatus is made of pyrex glass, so that it can be heated whenever there is a possibility of fractions solidifying and blocking the funnel.

Laboratory Stirrer. C. E. Waters. (*Ind. Eng. Chem.*, **1924, 16, 493**).—The stirrer described is operated by compressed air. A fan made of thin brass sheet and having twelve vertical vanes is fitted to the top of a short vertical steel shaft provided with bearings, and the lower end of this shaft is cemented on to a vertical glass tube forming the actual stirring rod. Small paddles, short closed side tubes, or the like may be attached to the bottom of this tube to aid the stirring action. Compressed air is blown on to the vanes from jets on a curved glass tube which is fixed horizontally and close to the outer edges of the vanes. W. P. S.

Reviews.

TECHNICAL METHODS OF CHEMICAL ANALYSIS. LUNGE AND KEANE. Second Edition. Edited by Charles A. Keane, D.Sc., Ph.D., and P. C. L. Thorne. M.A., M.Sc. Pp. xx.+702. London: Gurney & Jackson. Price 63s.

The first volume of the new edition of this work, which will be published in six separate volumes, has recently appeared. As stated in the preface, the volume under review contains two new sections, viz. "Electrolytic Methods of Chemical Analysis" and "Physical Methods employed in Chemical Analysis."

Section (1) of the new edition, "General Methods used in Chemical Analysis," written by Dr. Keane, is devoted mainly to the consideration of the fundamental operations of analytical chemistry. Whilst containing much that is common to all text books of analytical chemistry, the chapter is well and clearly written, but there are a few minor points which would bear revision. The description and illustration of a druggist's hand scales on p. 14 might well have been omitted, and one would have thought that the Gooch crucible was so well known as to need no description in a book written for chemists. In referring to the titration of boric acid on p. 43, the writer conveys the impression that glycerol produces a change in the indicator rather than in the acid, which is contrary to the generally accepted view. The statement occurs on p. 57 that "it is difficult to prepare a normal solution of barium hydroxide"; this compound is not sufficiently soluble in water at ordinary temperature to enable a *normal* solution to be made.

Of the new sections, that on "Electrolytic Methods of Chemical Analysis" is written by Dr. H. J. S. Sand, an original worker and acknowledged authority in this field. In the author's words, "this section consists of an account of the methods available for electrolytic analysis, of the apparatus employed, and the general conditions applicable to the solutions in which the electrolysis is effected." This section is a model of brevity and conciseness, and it is hoped that Dr. Sand will contribute further sections dealing with individual estimations.

The section on "Physical Methods employed in Technical Analysis," by Dr. J. S. G. Thomas, gives an account of many of the more important instruments employed in technical laboratories, and contains a great deal of valuable information which is not readily available to the majority of chemists. No mention is made of the spectrometer but, presumably, this will be included in a later volume.

The joint editors deal very fully with "Technical Gas Analysis," and the sampling and analysis of fuel is described by Mr. G. N. Huntly, who shows a first-hand knowledge of the subject treated.

The description of the methods used in the examination of the raw materials, products, and bye-products of the acid, alkali and chlorine industries, originally written by the late Dr. Lunge, has been revised for this edition by Dr. Dunn. Whilst acknowledging the debt which chemists owe to Dr. Dunn for the revision of this important section, the reviewer must confess to a feeling of disappointment in that the personal note is entirely lacking. A great variety of methods is included, but in far too many cases no opinion is expressed by the writer as to the value of the process described. It is noted that in the description of the determination of nitrate by reduction to ammonia and subsequent distillation the ammonia is collected in water, standard acid being added as the distillation proceeds. Surely it is more convenient to distil into excess of standard acid. In the description of the estimation of potash in saltpetre the perchlorate method is not mentioned.

The subject matter under the heading of pyrites includes the original Lunge's method together with others, and notably that of Allen and Bishop, whose method is extensively used in America. This latter method takes into account the influence of rate of addition of barium chloride upon occluded salts in barium sulphate, and is novel inasmuch as the initial attack is varied, whilst the final precipitation is made in the presence of iron in the reduced state. It is, however, erroneous to suggest that attack by means of nitric acid and potassium chlorate is accompanied by separation of sulphur. The methods given for the estimation of arsenic in pyrites include several which may, we hope without offence, be described as obsolete. The fusion method of Blattner and Brasseur yields only 80 per cent. of arsenic present, the missing 20 per cent. remaining in the residue after fusion; moreover, this method is not sufficiently delicate to yield accurate results when applied to minerals containing small amounts of arsenic. Undoubtedly the distillation method is the best available, but this cannot be considered to be presented in the most helpful fashion, for a much more simple preliminary treatment before distillation is now used in many technical laboratories. References to the work of Brand, Jannasch and Fenner would be of value in this connection.

A short description of pyrrhotite is given and could be well extended, together with references to work upon marcasite and pyrite, as the two former are frequently encountered in commercial deliveries of pyrites, and have significance as regards weathering in storage.

In the concluding section, Mr. H. Williams deals with the detection and estimation of cyanogen compounds in the various commercial products in which these occur. It is stated in the text that the methods selected are those which combine speed with accuracy, and the author appears to have succeeded in making a wise and discriminating choice. "Lunge" has always been regarded as a standard text-book upon the subject matter treated, but, with the present additions and revision, the book now becomes practically indispensable to all chemists who are engaged upon the general scope of the work; accuracy of description of methods is carried to a high degree and the new volume is to be recommended as worthy of being on the reference shelves of chemists, manufacturers and others who have to consider the subject from the technical aspect.

The indexing is ample and the references, on the whole, comprehensive.

G. RUDD THOMPSON.

X-RAYS. THEIR ORIGIN, DOSAGE AND PRACTICAL APPLICATION. By W. E. SCHALL. Pp. 119. Bristol: John Wright & Sons, Ltd. 1923. Price 5s.

It is common knowledge that X-ray therapy is frequently as efficacious as, and certainly preferable to a major operation for the treatment of certain diseases involving tissue degeneration. Probably thousands of exposures are made daily for diagnostic or curative purposes, so it is not surprising that severe injuries have occasionally been caused. Most of these are due to over-exposure or to excessive dosage. In view of the growing importance of the subject, the appearance of this little book, which bears evidence of having been written by one who has an extensive practical knowledge of the subject, must be welcomed. The author outlines in a simple way the theory of X-rays, then describes in fair detail the different kinds of tubes and other accessories required in connection with their application. The very important question of the quality of the rays, their measurement and dosage for diagnostic or therapeutic purposes is discussed in detail.

The reviewer ventures to call attention to two points which would increase the usefulness of the book in any subsequent edition; one is that, although the work of various investigators and their names are freely mentioned, in most instances no references are given to the books or journals in which the original papers may be consulted. This is of importance, especially in connection with certain methods of measurement for which full working details are necessary. The other point is that an index should be added; a table of contents at the beginning is by no means adequate for rapid reference.

H. E. COX.

TESTED METHODS OF MINERAL ANALYSIS. B. T. KITTO. Pp. 127. London: H. F. & G. Witherby. 1924. Price 7s. 6d. net.

A Foreword by Mr. Benedict Kitto states that the methods described in the present work are those that have been tested and found to give accurate results in his own laboratory, where the author has had twenty years' experience. The book is of a type with which, of late, metallurgical chemists have become rather familiar: it describes, in language of a rich local flavour and without any references to text-books or original papers, the routine followed in a particular laboratory.

In too many cases the chemical change occurring as the result of the operation prescribed is left unexplained, with the result that the less experienced operator has to follow such directions without understanding their bearing on the progress of the estimation.

The methods include a number that may now be considered out of date, while several are rather questionable. The following are examples of obsolescent processes. The nitrite method is used for the separation of nickel from cobalt, without the choice of an alternative. Dimethylglyoxime, nitroso-β-naphthol, and the volumetric cyanide method for nickel are not mentioned; nickel is estimated in the filtrate from the potassium cobaltinitrite precipitate by precipitation with potassium hydroxide (in a glass vessel) and ignition to oxide; no purification of the oxide from silica and adsorbed potassium salt is carried out. Similarly, the only "tested" method for total manganese is treatment of the filtrate from the basic acetate precipitation with sodium carbonate and ignition of the precipitate to Mn_3O_4, without subsequent purification. For the estimation of arsenic, the sulphide is converted into arsenic acid and the latter titrated with uranium acetate, ferrocyanide being the external indicator. With two iodimetric methods and an acidimetric method at his disposal, the operator can afford to discard his spot-plate when titrating for arsenic. The magnesium assay is carried out by titration of the magnesium ammonium phosphate with the same uranium solution as is used for arsenic; the magnesium value of the solution is obtained by calculation from the arsenic factor, clearly an error of judgment, as the end-point does not strictly correspond to the stoichiometric reaction. Sutton, one of the originators of the uranium method, insists that the standardisation should be done under exactly the same conditions as the assay. Cadmium is weighed as sulphide on a tared filter, by no means the best method; incidentally, the quantity of zinc ore taken is one grm., and the small precipitate of cadmium sulphide obtained therefrom collected on a 12·5 cm. filter; this tendency to use unduly large filter papers is fairly general throughout the book. For small quantities of titanium, the obsolete procedure of hydrolysing the dilute sulphuric acid solution is still recommended, despite the universal adoption of Weller's colorimetric method. As regards silica estimations, no mention is made of the final purity test by evaporation with hydrofluoric acid.

Hæmatite is wrongly credited with giving an iron-black streak (p. 54). Misprints are not numerous and are quite obvious, *e.g.* the long ton is stated to contain 32·6 ounces (p. 49).

In conclusion, the writer believes he voices the feelings of the great majority of his colleagues when he deplores the careless and loose style, the disregard of grammatical rules, and the use of slang in a growing number of technical books.

W. R. SCHOELLER.

THE VEGETABLE PROTEINS. By THOMAS B. OSBORNE, Ph.D., Sc.D. (Monographs on Biochemistry.) Pp. xiii.+154. London: Longmans, Green & Co. 1924. Price 9s. net.

Dr. Osborne's work on the vegetable proteins has gained him international repute, and we must welcome the appearance of the collective summary of his many researches on this very difficult subject. This book is obviously written by a specialist, and, as such, it has all the advantages and disadvantages of books which are written by highly specialised authors. The advantages of this book are many, but the disadvantages too are also striking. Thus about thirty years ago Osborne set out to disprove Ritthausen's theory that there is but one vegetable protein common to all plants. Since then Ritthausen has died and Osborne has become the recognised authority on plant proteins. In spite of this, Osborne continues to tilt against Ritthausen. The present state of the chemistry of the vegetable proteins reminds one of the chemistry of chlorophyll. Here, too, were two opposing schools which held that there were as many chlorophylls as plants, or that all plants contained the same chlorophyll respectively. This latter view has now been established beyond doubt by the classical researches of Willstätter. Is it therefore not possible that the plant proteins may some day also have their Willstätter to unite them?

English readers, for whom the Monographs on Biochemistry are, after all, mainly intended, will be disappointed to find that Osborne uses the American protein nomenclature. But still more bewildering is the fact that the "hæmoglobins" are classed among the "vegetable proteins" (p. 17). Similarly surprising is the statement that the protein from *Ceramium rubrum* belongs to the "hæmoglobins," since it "contains some derivative of *flavone*" (p. 32).

However, most disappointing is the incompleteness of the bibliography, although it covers 28 pages. Such important work as Guggenheim's on the protein and the amino acids of *Vicia faba* has been omitted, whereas three papers by Herzig on the methylation of several *animal* proteins with diazomethane are included. If it is intended to make special reference to this method of methylation of proteins, then, again, why have the publications of Biltz, Johnson and others been omitted? They also deal with the action of diazomethane on proteins and amino acids.

Reference must also be made to the omission of Zelinsky's work on the constitution of the proteins. It is true Zelinsky's results are fundamentally opposed to the now generally accepted concepts of Emil Fischer. But are we to be lulled into the belief that Emil Fischer was infallible? M. NIERENSTEIN.

REPORTS OF THE PROGRESS OF APPLIED CHEMISTRY ISSUED BY THE SOCIETY OF CHEMICAL INDUSTRY. Vol. VIII. Pp. 600. 1923. Price 7s. 6d. to members, 12s. 6d. to non-members.

In this exceedingly useful annual the sections are similar to those of previous issues, but the section on photographic processes is now omitted. No reason is given for the omission. Most chemists interested in photography will regret the loss of this section.

Marked advance is shown on the year's work in all branches of chemistry.

The section on Foods has again been written by Dr. G. W. Monier-Williams, and much of the analytical work on this subject published during the year will be found summarised here, as well as the work on Vitamins and Milk and Dairy Products. Other sections of interest to the public analyst are those on Oils, Fats, Waxes, by R. J. Pelly; Soils and Fertilisers, by H. J. Page; the Fermentation Industries, by H. L. Hind; and Sugar, Starch and Gums, by R. F. Lyle.

These annual volumes are indispensable to every chemist, and are worth possessing, if only for the full bibliographies under each section.

R. LEITCH MORRIS.

Publications Received.

CHEMICAL SYNTHESIS: STUDIES IN THE INVESTIGATION OF NATURAL ORGANIC PRODUCTS. By HARRY HEPWORTH. Pp. xx. + 244. London: Blackie & Son, Ltd. 1924. Price 20s. net.

CHEMICAL ENCYCLOPAEDIA. By C. T. KINGZETT. Pp. x. + 606. London: Baillière, Tindall & Cox. 1924. Price 30s. net.

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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Attempt to Extend Mitchell's Colorimetric Method to the Estimation of Catechol Tannins.

(Work carried out under the Analytical Investigation Scheme.)

By PHYLLIS HONOR PRICE, B.Sc.

(*Read at the Meeting, April* 2, 1924.)

MITCHELL'S colorimetric method (ANALYST, 1923, 48, 2) for pyrogallol tannins has been in constant use in this laboratory for nearly a year, and, in view of the very satisfactory results obtained with it, I have, at the suggestion of Dr. Nierenstein, attempted to extend this method to the catechol tannins. The results have been disappointing, probably owing to the behaviour of the catechol nucleus in forming abnormal complex iron salts. (*Cf.* Weinland, *Einführung in die Chemie der Komplex-Verbindungen*, 1919.)

Although it was found that the method is satisfactory for the comparison of (1) Catechol with Catechol, (2) Protocatechuic Acid with Protocatechuic Acid and (3) Catechin with Catechin, yet the ratios which were obtained, on comparing these three substances with one another, were most unsatisfactory. This is shown by the results, some of which are given below. Unless otherwise stated, they were obtained from experiments carried out similarly to those described by Mitchell for the pyrogallol tannins, *i.e.* one c.c. of a 0·1 per cent. solution of the substance to be investigated was put into about 95 c.c of water in a Nessler tube provided with a Hehner's side tubulure and tap, 2 c.c of the reagent were added, and the solution was made up to 100 c.c. and compared with another solution prepared in like manner.

The water was estimated in each substance used and allowed for in the calculated ratios. These were obtained by taking the molecular weight ratios.

COMPARISON OF CATECHOL AND PROTOCATECHUIC ACID.—The calculated ratio is as follows:

$$\frac{\text{Catechol, } C_6H_4(OH)_2, \quad \text{M.W. } 110}{\text{Protocatechuic acid, } C_6H_3(OH)_2.COOH+H_2O, \quad \text{M.W. } 172} = \frac{110}{172} = \frac{1}{1\cdot56}.$$

Calculated ratio in c.c. =64:100. Found ratio in c.c. =88·9:100, 88·1:100, 89·9:100, 88·8:100, 89·6:100, 89·9:100, 89·5:100, *93·7:100, 89·4:100, *94:100, 91·1:100 and *89:100.

Quite freshly prepared solutions, which standing could not have affected, gave the following ratios:—100:100, *100:96·3, *100:100 and 100:92·3.

A 0·1 per cent. catechol solution was compared with a 0·4 per cent. protocatechuic acid solution and the latter was diluted until the colours could be matched. Found ratio in c.c. =89·2:100, 89·5:100, 89·6:100, 90·1:100, 91·5:100, 89·5:100, 87·7:100 and 89·8:100, when calculated for 0·1 per cent. solutions.

It is evident, therefore, that the ratio found when comparing catechol with protocatechuic acid is not a molecular one, as observed by Mitchell in the case of pyrogallol and gallic acid, where the carboxyl group acts as a diluent.

CATECHOL AND GALLIC ACID.—

Catechol, $C_6H_4(OH)_2$, M.W. 110
Gallic acid, $C_6H_2(OH)_3.COOH+H_2O$, M.W. 188

$$\frac{110}{188}=\frac{1}{1\cdot71}.$$

Calculated ratio in c.c. =58·5:100. Found ratio in c.c. =100:57·1, 100:57·1, 100:56·5, 100:57·6, 100:58·5, *100:44·8, 100:55·3, *100:52, 100:58 and *100:61.

For a 0·1 per cent. catechol solution compared with a 0·4 per cent. solution of gallic acid the found ratio in c.c. =100:56, 100:51·3 and 100:50 calculated for 0·1 per cent. solutions.

It should be noted that the ratio found is the reciprocal of the calculated one, which is strange.

PROTOCATECHUIC ACID AND GALLIC ACID.—

Protocatechuic acid, $C_6H_3(OH)_2.COOH+H_2O$, M.W. 172
Gallic acid, $C_6H_2(OH)_3.COOH+H_2O$, M.W. 188

$$\frac{172}{188}=\frac{1}{1\cdot093}.$$

Calculated ratio in c.c. =91·5:100. Found ratio in c.c. =100:57, *100:57, 100:61, *100:56, 100:55, *100:50, 100:51·9, *100:51·9, 100:51·9, 100:50·5, 100:51, *100:51·9, *100:51, 100:58·5, 100:60·7, and 100:59·4.

CATECHOL AND CATECHIN.—

Catechol, $C_6H_4(OH)_2$, M.W. 110
Catechin, $C_{15}H_9O(OH)_5+3H_2O$, M.W. 344

$$\frac{110}{344}=\frac{1}{3\cdot126}.$$

Calculated ratio in c.c =100:312·5 Found ratio in c.c. =100:213, 100:213, 100:199, 100:200, 100:195, 100:199, 100:200, 100:197, 100:193, 100:191, 100:213, 100:207, 100:210, 100:205, 100:211, and 100:213.

EXPERIMENTS WITH STANDARD SOLUTIONS AND MIXTURES.—According to the work on the pyrogallol nucleus, normal solutions of different substances should match each other if they behave similarly.

A 0·01 *N* solution of catechol was therefore compared with a 0·01 *N* solution of anhydrous catechin.

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* **These readings were taken by Miss W. N. Nicholson, B.Sc.**

Calculated ratio in c.c. = 100:100. Found ratio in c.c. = 100:72·5, 100:72·4, 100:75·4, 100:67·4, 100:75·6, 100:75·5, 100:72·0, 100:73·5, 100:74·1, 100:75·0, and 100:74·6.

Equal volumes of 0·1 per cent. solutions of gallic and protocatechuic acids were mixed, and the resulting solution was compared first with gallic acid, and then with protocatechuic acid. The calculated ratios for these experiments were obtained from the one found above for protocatechuic and gallic acids.

$$\frac{\text{Gallic acid}}{\text{Protocatechuic acid}} = \frac{57{\cdot}3}{100} = \frac{1}{1{\cdot}744}.$$

$$\text{Equal volumes mixed} = \frac{1+1{\cdot}744}{2} = 1{\cdot}372.$$

$$\text{Therefore} \quad \frac{\text{Gallic acid}}{\text{Mixture}} \quad \frac{1}{1{\cdot}372}.$$

Calculated ratio in c.c. = 74·8:100. Found ratio in c.c. = 73·0:100, 71·62:100, 74·11:100, 77·27:100, 73·16:100, 73·86:100, 72·88:100, and 74·0:100.

It is of interest to note that these values closely approach those required by theory, if the ratio chosen from which to calculate the theoretical result was correct.

$$\frac{\text{Protocatechuic acid}}{\text{Mixture}} = \frac{1{\cdot}744}{1{\cdot}372} = \frac{1{\cdot}27}{1}.$$

Calculated ratio in c.c. = 100:78·7. Found ratio in c.c. = 100:71·79, 100:72·05, 100:71·22, 100:68·0, 100:72·0, 100:71·34, 100:66·59, and 100:67·65.

$$\frac{\text{Pyrogallol, } C_6H_3(OH)_3 + 4{\cdot}6 \text{ per cent. } H_2O, \quad \text{M.W. } 132}{\text{Catechol, } C_6H_4(OH)_2, \quad \text{M.W. } 110} = \frac{132}{110} = \frac{1{\cdot}2}{1}.$$

Calculated ratio in c.c. = 100:83. Found ratio in c.c. = 41:100, 40:100, 42:100, 45:100, and 43:100.

These readings were carried out jointly with Miss Nicholson. The last ratio was obtained with quite freshly prepared solutions.

A 0·01 *N* solution of pyrogallol was compared with solutions of catechol of various concentrations. The ratios varied as shown below. Since fresh pyrogallol solutions were not prepared each time, the variations are probably a measure of the oxidation of the pyrogallol nucleus. The results cannot, however, be used as a basis for calculation, since the readings were not taken at definite intervals.

$$\frac{\text{Pyrogallol}}{\text{Catechol}}.$$

0·01 *N*:0·01 *N*. Expected ratio = 8:8. Found ratio = 3:8.

The following calculated ratios were obtained from the above found ratio:—

0·01 *N*:0·015 *N*.	Calculated ratio = 4·5:8.	Found ratio = 6:8.
0·01 *N*:0·02 *N*.	Calculated ratio = 6:8.	Found ratio = 8·8:8.
0·01 *N*:0·04 *N*.	Calculated ratio = 12:8.	Found ratio = 22·2:8.

The ratio 3:8 for 0·01 N solutions is theoretically 45:100 for 0·1 per cent. solutions as was found in one case given above. For pyrogallol against catechol the ratio 3:8 seems strange, since the ratio of the hydroxyl groups is 3:2. From these results, therefore, I am inclined to assume that 1 molecule of pyrogallol corresponds to 4 molecules of catechol.

From the above results it will be seen that with different substances the same ratio was rarely obtained twice following.

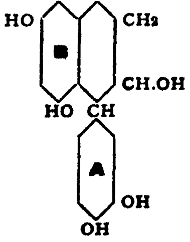
In each case the violet colorations had essentially to be of the same shade, or as nearly as possible the same, before the intensities could be compared. Many preliminary experiments were necessary for such an adjustment. Until all the substance which was present containing the catechol nucleus had been converted into the soluble ink, the addition of the reagent caused the amount of the violet colour to increase and thus the solution in the cylinder to darken, but once a sufficient quantity had been added, the addition of even a few drops more of the reagent turned the violet from a blue to a pink shade, probably due to the yellow colour of the reagent. With one solution slightly bluer than the other it was not easy to make correct estimations. The solutions were viewed vertically by daylight and by artificial light. If viewed horizontally, they looked very unlike.

The fact that the ratios $\frac{\text{Catechol}}{\text{Gallic acid}} = \frac{100}{57}$ and $\frac{\text{Protocatechuic acid}}{\text{Gallic acid}} = \frac{100}{57}$ were obtained is additional evidence showing that the carboxyl group in protocatechuic acid does not dilute the catechol nucleus.

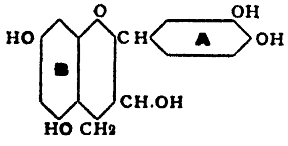
Possible Explanation of the Ratios.—Attempts were made to find explanations for the ratios obtained between the different substances in terms of the numbers of hydroxyl groups as well as the molecular weights. They were not, however, a success. This was also confirmed by the series of experiments kindly carried out by Miss Nicholson.

When comparing catechol with catechin it was found that the ratios varied very considerably according to the length of time the catechol solution had been standing. Fresh solutions, therefore, were absolutely necessary.

Experiments showed that a 0·01 N solution of catechin gives rise to a darker violet colour than a 0·01 N solution of catechol. Since catechin, according to the constitutional formula assigned to it by Nierenstein (*J. Chem. Soc.*, **1920, 117,** 971), and also according to the Perkin and Freudenberg formula (*J. Chem. Soc.*, **1905, 87, 404**; *Ber.*, **1920, 53,** [B], 1416), contains the catechol nucleus A and the



Nierenstein's Formula.



Perkin and Freudenberg's Formula.

resorcinol nucleus B (which on opening in solution may lead to the formation of the phloroglucinol nucleus), it was thought that probably the additive effect between the nuclei A and B causes the darker colour. Attempts were therefore made to elucidate this point. Catechol was placed in each cylinder and resorcinol was added to one. Even 10 c.c. of 0·01 *N* resorcinol gave no change. Phloroglucinol acted similarly. Therefore the darker colour produced with the reagent by catechin is not due to the additive effect of the resorcinol or phloroglucinol nuclei.

THE GREEN COLORATION IN ACID SOLUTION.—A series of experiments was also carried out to find whether the catechol nucleus could be estimated in acid solution by using the green coloration produced.

The violet-coloured solutions were prepared as usual, and a small quantity of an acid solution (10 c.c. of glacial acetic acid in 90 c.c. of water) was added.

Protocatechuic acid	+0·3 c.c of acid produced an apple-green colour.
Catechol	+0·3 c.c. of acid produced a yellowish-green colour.
Gallic acid	+1 c.c. of acid produced a greyish-green colour.
Pyrogallol	+1 c.c. of acid produced a greyish-green colour.

The green colour was very faint and of no use for comparisons. On diluting with tap water it turned bluish owing to the alkali in the water. When the solutions in the cylinders were made up with distilled water, and acid was added, a pale yellow colour was formed, but part of this was due to the yellow reagent. It seemed possible to tell by the shade of yellow whether any substance containing the pyrogallol nucleus or the catechol nucleus was present or not, and if so, which, but the amount could not be estimated.

EFFECT OF QUININE HYDROCHLORIDE.—In estimating gallotannin in the presence of gallic acid Mitchell uses quinine hydrochloride to precipitate the tannin. The most serious objection to this method for catechol tannins is, however, the fact that the presence of quinine hydrochloride (which is absolutely essential) with the reagent in a solution of catechol, protocatechuic acid or catechin causes precipitation. This does not occur with (1) the substance and quinine hydrochloride, (2) the substance and the reagent, or (3) the reagent and quinine hydrochloride, but only when all three are present together. Cinchonine sulphate solution acts similarly. On adding it to the violet solution a milky precipitate comes down immediately, which, in a few minutes, takes all the colour from the solution. On filtration a clear colourless filtrate is obtained, and a violet-coloured residue is left on the filter paper.

This definitely shows that the method cannot be applied to catechol tannins.

SUBSTANCES GIVING THE VIOLET COLORATION.—Mitchell, in his paper, says: "The reagent does not give any coloration with phenol or other mono-hydroxylated compounds, nor with salicylic acid or other compounds containing two hydroxyl groups. So far as my observations go, the violet colour is specific for the pyrogallic grouping."

It is interesting to note from a series of experiments which I have carried out that two hydroxyl groups in the *ortho* position are always necessary to get the violet coloration. Of the following substances investigated: phenol, hydroquinone, resorcinol, phloroglucinol, salicylic acid, meta-hydroxy-benzoic acid, β-resorcilic acid and guaiacol do not give the violet colour, whereas, catechol, protocatechuic acid, pyrogallol, gallotannin and gallic acid do give the violet colour.

BIOCHEMICAL LABORATORY,
UNIVERSITY OF BRISTOL.

Estimation of Sugar in Urine by Means of Fehling's Solution with Methylene Blue as Internal Indicator.

BY J. HENRY LANE, B.SC., F.I.C., AND LEWIS EYNON, B.SC., F.I.C.

(*Read at the Meeting, May* 7, 1924.)

ABOUT a year ago we proposed the use of methylene blue as an internal indicator for Fehling's solution, and described a method of procedure for the exact volumetric determination of the more important reducing sugars (*J. Soc. Chem. Ind.*, 1923, 32T.; ANALYST, 1923, **48**, 220). For sugar products we have found the method entirely satisfactory, but the case of urine stands somewhat apart, inasmuch as the greater part, if not the whole, of the reducing power of normal urine towards Fehling's solution is due to substances other than sugar, *e.g.* creatinine and uric acid. Methylene blue by itself in alkaline solution has been proposed for the detection of sugar in urine, although, according to Wender (*Chem. Zeit.*, 1893, 670), it is reduced by creatinine. Allen pronounced it very useful as a qualitative or limit test, but too sensitive to back-oxidation for exact quantitative purposes (*Chemistry of Urine*, p. 82). We find no reference to its use as an indicator for Fehling's solution, although the fact that it is decolorised by non-sugars present in urine would not necessarily impair its indicator action, provided that it retained its colour until all copper had been reduced.

If normal untreated urine is titrated against 10 c.c. of Fehling's solution in presence of methylene blue, by the incremental method described in our earlier paper (*loc. cit.*), no satisfactory end-point is obtained, because before all the copper is reduced the alkalinity of the Fehling's solution is so much diminished by the natural acidity of the added urine that the indicator cannot function properly. This difficulty can be overcome by first rendering the urine neutral to litmus. Operating on these lines with healthy urines we have found that 10 c.c. of Fehling's solution require about 40 to 70 c.c., corresponding to 0·12 to 0·07 per cent. of reducing matter calculated as dextrose, in the samples. In these titrations we found it advisable to use 10 drops of the 1 per cent. methylene blue solution. The greater part of the colour of the indicator is discharged before the end-point is reached, but it is the disappearance of the last trace of greenish shade which marks the complete reduction of the copper, and after some practice this point can be determined to within 5 c.c.

In order to obtain a sharper end-point with these healthy urines we have tried defecation with normal lead acetate. This substance is widely used for the defecation of sugar products, since, unlike basic lead acetate, it does not remove any sugar from solution (*cf. J. Soc. Chem. Ind.*, **1923**, **463**T.; ANALYST, **1924**, **49**, **90**). It is necessary to remove the excess of lead from the defecated liquid, and for this purpose potassium oxalate is very suitable. To avoid dilution of the urine the reagents may be used in the dry state, the neutralised urine being first shaken for a minute or two with 3 grms. of powdered normal lead acetate per 100 c.c., then filtered through a dry filter and shaken for a minute with 1 grm. of powdered potassium oxalate per 100 c.c., and again filtered. Experiments on urines have shown that this treatment does not remove any reducing sugar. It does remove a small quantity of reducing non-sugars, equivalent in reducing power to about 0·02 to 0·03 per cent. of dextrose, so that the percentage of sugar found after defecation will be nearer by this amount to the true value than that found without defecation. Such a small difference, however, is practically negligible, and the only advantage of defecation with normal lead acetate is that it renders the urine much lighter in colour and makes the end-point of the titration more distinct. This is an advantage in the titration of urines of low reducing power, corresponding to about 0·2 per cent. or less of dextrose, but when the reducing power rises to values of pathological significance, and the urine has to be diluted before titration against 10 c.c. of Fehling's solution, the end-point can be recognised without difficulty, whether the urine is defecated or not. The higher the percentage of sugar in a urine, the more does it need to be diluted before titration, and the greater becomes the precision of the titration, since the interfering non-sugars are correspondingly diluted. As the sugar-content of the urine rises beyond 1 or 2 per cent., the degree of precision approaches that attainable with pure sugar solutions, which is of the order of 0·2 per cent. of the amount of sugar estimated.

On the basis of these observations we propose the following method of testing urines for sugar. It has the advantage of starting with a sort of limit test, which is very simple to carry out, and beyond which it is not necessary to go if the reducing power corresponds with that of normal urines. It is essential that the sample should first be rendered neutral if it is acid; addition of 1 c.c. of a 10 per cent. solution of sodium hydroxide per 100 c.c. of urine is usually sufficient, and, if necessary, the sugar-content found must be corrected for this dilution. Defecation with normal lead acetate may be carried out as described above, but is not essential. Ten c.c. of Fehling's solution (Soxhlet's modification, containing 34·64 grms. of crystallised copper sulphate, 173 grms. of Rochelle salt and 50 grms. of sodium hydroxide per litre) in a flask of about 300 c.c. capacity are treated with 20 c.c. of the neutralised urine and heated to boiling over a wire gauze. It is a good plan to introduce a trace of vaseline on the end of a glass rod into the liquid, to prevent excessive frothing at a later stage. When the liquid has been boiling for a minute 10 drops of a 1 per cent. aqueous methylene blue solution are added (preferably without touching the sides of the flask) and boiling is continued. If the methylene blue is completely decolorised within about 15 seconds, so that the reaction liquid appears exactly as before the addition, the urine contains more than 0·25 per cent. of reducing matter calculated as dextrose, and to estimate the amount a fresh titration must be carried out with the urine suitably diluted. If, on the other hand, as is usually the case with normal urines, the methylene blue is not completely decolorised, the amount of reducing substances is less than 0·25 per cent., and, if it is necessary to estimate their amount, the titration is continued by adding 10 or 5 c.c. increments of urine to the boiling liquid in the flask, with about 15 seconds' actual ebullition after each addition, until all trace of greenish tint has disappeared. For the estimation of such small quantities of reducing substances it may be taken that 10 c.c. of Fehling's solution correspond to 0·05 grm. of dextrose.

In cases where this preliminary test shows more than 0·25 per cent., and the titration has to be repeated after suitable dilution of the sample, the procedure described in our earlier paper (*loc. cit.*) may be followed. Ten c.c. of Fehling's solution are treated with 15 c.c. of the diluted urine in a flask of about 300 c.c. capacity and heated to boiling over a wire gauze, and further additions of urine are made at intervals, with about 15 seconds' actual ebullition after each, until almost all the copper appears to be reduced, whereupon 3 to 5 drops of the indicator solution are added, and the addition of urine is continued in small increments until the indicator is completely decolorised. If the maximum accuracy is desired, it is advisable to repeat the titration by our standard method (*loc. cit.*), 10 c.c. of Fehling's solution being boiled for 2 minutes with almost the whole volume of diluted urine required for its reduction, after which 4 drops of indicator are added and the titration is completed in 1 minute further. The results are interpreted by means of the dextrose table given in our earlier paper and reproduced here (Table I.).

TABLE I.

Dextrose Table for 10 c.c. of Fehling's Solution.

Sugar solution required. c.c.	Anhydrous dextrose corresponding to 10 c.c. of Fehling's solution. Mgrms.	Anhydrous dextrose per 100 c.c. of sugar solution. Mgrms.
15	49·1	327
16	49·2	307
17	49·3	289
18	49·3	274
19	49·4	260
20	49·5	247·4
21	49·5	235·8
22	49·6	225·5
23	49·7	216·1
24	49·8	207·4

TABLE I.—(*continued*).

Sugar solution required. c.c.	Anhydrous dextrose corresponding to 10 c.c. of Fehling's solution. Mgrms.	Anhydrous dextrose per 100 c.c. of sugar solution. Mgrms.
25	49·8	199·3
26	49·9	191·8
27	49·9	184·9
28	50·0	178·5
29	50·0	172·5
30	50·1	167·0
31	50·2	161·8
32	50·2	156·9
33	50·3	152·4
34	50·3	148·0
35	50·4	143·9
36	50·4	140·0
37	50·5	136·4
38	50·5	132·9
39	50·6	129·6
40	50·6	126·5
41	50·7	123·6
42	50·7	120·8
43	50·8	118·1
44	50·8	115·5
45	50·9	113·0
46	50·9	110·6
47	51·0	108·4
48	51·0	106·2
49	51·0	104·1
50	51·1	102·2

The results obtained with the method described above agree closely with those given by Gerrard's cyano-cupric method, but are considerably lower than those obtained with Pavy's method. The higher results given by the latter method are to be attributed to the non-sugars, as is indicated by the figures in Table II.

TABLE II.

Sugar Estimations in Urine.

By means of Fehling's Solution with Methylene Blue Indicator.				By means of Pavy's Solution.			
Original reducers as dextrose R. Per Cent.	Added dextrose D Per Cent.	R + D	Total found. Per Cent.	Original reducers as dextrose R′. Per Cent.	Added dextrose D. Per Cent.	R′ + D	Total found. Per Cent.
0·12	0·10	0·22	0·21	—	—	—	—
0·09	0·29	0·38	0·38	0·17	0·29	0·46	0·47
0·09	0·57	0·66	0·67	0·17	0·57	0·74	0·75
0·10	1·45	1·55	1·59	0·19	1·45	1·64	1·64
0·10	2·90	3·00	3·00	0·19	2·90	3·09	3·27

referring to comparative estimations by Pavy's and our methods. All the figures in the same horizontal line relate to the same sample of normal urine, neutralised, defecated with normal lead acetate and freed from lead by means of potassium oxalate, without and with addition of a known quantity of dextrose. The Pavy's solution used contained per litre: 4·158 grms. of crystallised copper sulphate, 20·4 grms. of Rochelle salt, 20·4 grms. of potassium hydroxide, and 300 c.c. of concentrated ammonia; 40 c.c. were used for each titration and taken as equivalent to 0·02 grm. of dextrose.

It is clear from the table that Pavy's method gives higher values than ours for the reducing substances of normal urine, and as these substances consist chiefly, if not entirely, of non-sugars, this is a point in favour of the new method. Allowing for these differences in the original reducer-content, the results for the added dextrose are practically the same by both methods. For large amounts of sugar our method is much more accurate than Pavy's, since the end-point can be determined with greater precision by the former. In the estimation of sugar in urine, however, extreme accuracy is, in many cases, not essential, and the chief practical advantages of the new method over Pavy's are its greater convenience and cheapness, and the avoidance of ammoniacal vapours.

It may be of interest to mention that methylene blue can be used as an internal indicator in Pavy's solution. It does not act quite so readily as it does in Fehling's solution, but it sharpens the end-point, especially in the case of coloured liquids. If it were employed in this way for accurate work, it would be necessary to add always the same amount of indicator and to standardise the method against dextrose, as the end-point, although fairly sharp, comes somewhat later than when Pavy's solution is used without an indicator.

DISCUSSION.

Mr. A. **CHASTON CHAPMAN** said that, speaking from his own experience, he was inclined to give preference to the method of Pavy.

Mr. **WM. PARTRIDGE** said that accuracy in sugar estimations was very important in cases of diabetes where patients were being kept alive largely by the skill in dieting by the doctor. In such cases the useful figure was, not the percentage, but the actual amount, of sugar passed per diem. In his opinion Fehling's solution was not always reliable for urine, but was most useful when used gravimetrically. The most accurate method of estimating sugar in urine was to take the gravity, ferment with washed yeast, and take the gravity again.

Mr. C. L. **CLAREMONT** enquired whether the authors found differences in samples of methylene blue. In his experience the German product contained a good deal of methylene mauve. He asked whether there were different varieties of methylene blues, as it seemed to him they varied with different firms.

Mr. **JOHNSON** asked the authors it they would give their reasons for using this special table of equivalents for their estimations.

Mr. G. R. **THOMPSON** thanked the authors for their novel contribution and asked whether they had experimented with this method with abnormal urine. In his opinion, the potassium iodide method was a good one, in that with morphine present there would be no interference. He considered the Fehling's solution method a more desirable one than Pavy's method.

Mr. L. EYNON, in reply, said that the gravimetric method suggested by Mr. Partridge was slow and troublesome, and not so good as the volumetric method with an internal indicator. They had not found variation in samples of methylene blue, such as Mr. Claremont mentioned, although one sample had been unsatisfactory. The table in the paper, referred to by Mr. Johnson, was one published in the Journal of the Society of Chemical Industry, which had been prepared with great care and gave good results. They had not tried any abnormal samples of urine; they had worked with normal urine to which dextrose had been added. The effect of morphine had not yet been worked out, and they were unable to say what it would be.

The Estimation of Copper and Tin in Copper-Tin Alloys.*

BY A. T. ETHERIDGE, M.B.E., B.Sc., F.I.C.

(*Read at the Meeting, May* 7, 1924.)

THE common method of separating tin as stannic oxide by the action of nitric acid has many disadvantages. The stannic oxide occludes practically every other metal in the alloy, as well as phosphorus from phosphor bronze, and the total amount of impurity may be from 5 to 10 per cent. It has been found well-nigh impossible to decompose the impure oxide at one fusion, the difficulty apparently increasing with increase of temperature of ignition. Moreover, the assumption that the sulphides, after ignition, can be considered to be oxides is not correct, and introduces an error in the estimation of tin by difference; also the separated sulphides are apt to retain tin, which is difficult to wash out, and leads to low results if the attempt is made to estimate tin directly. It has also been found that the crucibles, necessarily of porcelain, are severely injured by the fusion, even when carried out carefully over a small flame. Variations in the initial nitric acid attack have been suggested as a means of diminishing occluded impurities, but, in my experience, these have not produced a pure stannic oxide.

The special case of phosphorus from phosphor bronze is well known. It is assumed that all the phosphorus is present in the precipitate as P_2O_5, and an allowance is made after the phosphorus has been estimated; or, alternatively, the tin is estimated directly as mentioned above.

For these reasons the author has long discarded the nitric acid treatment in favour of a new method worked out after numerous experiments. The principle of the new method is to separate copper from a sulphuric acid solution by electrolysis, the conditions being such that the tin is held completely in solution, and is not precipitated by hydrolysis. This method of separation is not referred to in any books dealing with electro-analysis, as apparently it has not been realised that copper can be separated quantitatively from strongly acid solutions.

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* Communication from the Research Department, Woolwich.

The details of the method are as follows:—One grm. of the alloy (or 0·5 grm. if the tin exceeds 10 per cent.), preferably drillings or turnings (freed from iron particles by a magnet) is dissolved in a mixture of 60 c.c. of dilute sulphuric acid (one vol. of conc. acid to three vols. of water) and 5 c.c. of nitric acid (sp. gr. 1·42) in a 400 c.c. squat beaker. If the solution is quite clear it is diluted to 200 c.c. and electrolysed. If a gelatinous residue is observed it is an indication of phosphorus or arsenic or both, probably as stannic phosphate or arsenate. If lead is present a dense white precipitate of lead sulphate will be observed, and this masks any other residue which may be there. In either case it is necessary to evaporate the solution carefully until it fumes, after adding about 20 c.c. more dilute sulphuric acid, to allow for sulphuric acid lost by evaporation. This treatment decomposes the stannic phosphate (or arsenate) and also allows lead to be quantitatively separated in the next operation. After cooling down to room temperature, about 150 c.c. cold water are quickly added, and the liquid is well shaken, to prevent local overheating. Excessive heating would cause separation of tin as hydrate by hydrolysis, and this would necessitate evaporating again or starting afresh. A clear liquid at this stage indicates absence of lead. Any lead sulphate separates rapidly as a heavy white precipitate and can be filtered off after the beaker has stood for about 30 minutes in cold running water. The lead is estimated in the usual way. If it is small in amount, say not more than 2 or 3 per cent., there is no danger of loss of copper or tin by occlusion. After the lead sulphate has been washed the volume of the filtrate is approximately 200 c.c. and, having been caught in a 400 c.c. beaker, is ready for electrolysis after the addition of 10 c.c. of nitric acid (sp. gr. 1·2) or 5 c.c. of colourless nitric acid (sp. gr. 1·42). The addition of nitric acid in small amounts is necessary in order to obtain a good copper deposit in the subsequent electrolysis. Of course no further nitric acid need be added if the alloy dissolves completely in the initial acid mixture. If tin separates by hydrolysis on standing, or during electrolysis, it is an indication of insufficient sulphuric acid, and a fresh start must be made, more acid or less alloy being used, or more allowance made for evaporation.

SEPARATION OF COPPER BY ELECTROLYSIS.—A rotating cathode of platinum gauze is used with a small platinum foil anode. There is nothing quite so satisfactory as platinum for the cathode. The gauze form is not absolutely essential, but it is necessary to have the cathode rotating (or the liquid stirred) on account of the heavy currents used. If the cost is prohibitive platinum might be replaced by silver. In that case the copper is subsequently removed by an ammonia solution of trichloracetic acid (*J. Amer. Chem. Soc.*, 1921, 43, 700). A platinum anode is essential, but is so small that it is comparatively inexpensive. A current of 4 amps. is used, from a 10 or 20 volt circuit, provided with a rheostat of such a resistance that the current can be lowered to 0·5 amp. or less. After the current has been on 30 minutes, about 0·5 grm. of urea is added to destroy any nitrous acid developed by the current. (Nitrous acid tends to delay the copper deposit.) When the liquid has become apparently free from copper the same current is continued for a further 30 minutes. Hydrogen is often generated at this stage in large volumes, but has no effect on the deposit. At the end of this time the current is reduced by 0·5 amp. every 15 minutes, down to the final stage of 0·5 amp. At each change 0·5 grm. of urea is added.

The sides of the beaker and the exposed platinum are washed from time to time with the wash-bottle jet, and finally the beaker is lowered away from the cathode, with the current left on, and the cathode washed at the same time with the wash bottle jet. The cathode is now detached, washed with alcohol, dried in a low-temperature oven, and weighed. The initial treatment of the cathode, after ignition in a muffle, having been the same, the difference in weight represents the copper in the alloy weight taken.

The cathode must not be allowed to stand wet in the air. It should be dipped into alcohol as soon as it is washed. A good colour of the deposit indicates freedom of the alloy from antimony and bismuth, which metals are, to some extent, deposited with copper. Unusually large amounts of either would completely blacken the copper and render the method useless, but traces, which cause slight discoloration only, can be ignored. Bronzes of high quality give an extremely pure copper, whilst ordinary commercial bronzes of medium quality nearly always give some discoloration. If the discoloration is too bad to be ignored the analyses would have to be abandoned and the alloy treated in an entirely different way, as described under the heading of analysis of copper-antimony (or bismuth)—tin alloys. The author has not come across any bronzes of this kind, however, and the possibility of encountering them appears to be remote.

ESTIMATION OF TIN.—The anode is now washed and removed. (Slight traces of lead peroxide may occasionally be observed on the anode, usually from alloys which have dissolved completely in the initial acid mixture, indicating traces of lead in the alloy). The tin is precipitated as stannic sulphide by the passage of a rapid current of hydrogen sulphide about 30 minutes. The precipitate is allowed to stand some time, preferably overnight, before filtering. It is best filtered through ashless pulp, and washed with dilute ammonium nitrate solution. Washing with water tends to carry some of the precipitate through the filter.

The precipitate is transferred to a porcelain crucible, previously covered with a layer of ashless paper, to prevent contact of the sulphide with the porcelain.

Ignition is carried out for some time at a low heat (below dull red), preferably first in a low temperature muffle, and is afterwards completed in a high temperature muffle at the usual red heat. In this way stannic sulphide can be completely converted into oxide. The precipitate is brushed out, when cold, into a counterpoised aluminium scoop and weighed.

Unless the precipitate is unusually large it is not contaminated with iron or any other metals, such as zinc or nickel. These are easily washed out, but, if the precipitate is too large, it is better to start again with 0·5 grm. of alloy. Contamination with antimony and bismuth is possible, as these are only partly deposited with copper, but their presence will have been detected, as mentioned, by the colour of the copper deposit. Traces can be ignored.

Cadmium is seldom present in bronzes, but is sometimes met with in brasses, *e.g.* Admiralty brasses with 1 per cent. tin. These alloys (of which 2 grms. is a convenient weight to use, with 7 c.c. of 1·42 nitric acid instead of 5 c.c.) might contain cadmium which would contaminate the tin, although the author has not met with such a case. If it is present to the extent of, say, 0·1 per cent., the precipitate of stannic sulphide should be extracted with sodium sulphide, filtered after standing, and the tin reprecipitated.

Arsenic may be in the solution and is then precipitated with the tin. It appears, however, to be completely volatilised by the method of ignition described. No interference with the method has been observed in the case of bronzes containing 0·5 per cent. of arsenic, most of which may, conceivably, be removed as hydrogen arsenide at the cathode, during electrolysis. It is not deposited on the cathode under the conditions used in this process.

The complete removal of copper from solution by the method given is certain. If any black turbidity is observed when the hydrogen sulphide is first introduced it is an indication that copper is present and that the conditions for electrolytic separation have not been correct, and a fresh start must then be made.

The filtrate from the stannic sulphide is used for estimation of iron, nickel, zinc, etc. Alternatively the filtrate can be used for the estimation of phosphorus, if desired, but this is seldom convenient since 2 grms. is usually the best amount to take. Also, it is quicker to separate copper and tin together as sulphide from hydrochloric acid solution in one operation, and to use the filtrate for the estimation of phosphorus.

DISCUSSION.

Mr. J. MYERS, referring to a remark in the early part of the paper that stannic oxide occluded every other metal, said that this was undoubtedly a source of error in the usual method of separating tin. The author had devised a method which involved a sharp separation, and he would like to congratulate him on a serious and successful effort to overcome a constantly occurring difficulty in metallurgical analysis.

He had looked with some suspicion upon the separation of tin with hydrogen sulphide in the presence of nitric acid, which had been generally considered an unsafe method, but, upon making trial with similar solutions both in presence and absence of nitric acid, and also in presence of arsenic, his results were all in excellent agreement with control tests, where tin was reduced with antimony and titrated with iodine. He was satisfied on this point and very much pleased with the method. If the author had details of results obtained by one or other of the well known volumetric methods applied to tin in a sample of bronze, together with results by the new method, the figures would be both interesting and of value in furnishing a comparison of the degree of accuracy which could be obtained by the older methods.

Mr. W. H. WOODCOCK said he was much interested in the paper, but was surprised to hear this was put forward as a new process, for it was one he had used for over 20 years. Regarding the problem of current for electro-deposition, it was possible to get over the difficulty by using an ordinary power main with a lamp resistance, and leaving it running all night; copper could be obtained by this means quite pure; it was usual to oxidise tin sulphide to stannic acid.

Mr. G. RUDD THOMPSON said that, in his opinion, it was absolutely necessary to use platinum for the electrolytic deposition of copper, but silver could be used with other metals.

The Estimation of Ammonia by Means of Sodium Hypobromite.

By MAXWELL BRUCE DONALD, M.Sc., A.R.C.Sc., A.I.C.

EARLY workers in this field generally used brominated solutions of sodium hypochlorite; these are not only difficult to prepare accurately, but also lead to the formation of by-products such as hydrazine, J. Thiele (*Annalen*, 1893, **273**, 160), and possibly nitrogen trichloride, W. A. Noyes and A. C. Lyon (*J. Amer. Chem. Soc.*, 1901, **23**, 460). The use of sodium hypobromite appears to have been first suggested by W. Knop (*Zeitsch. anal. chem.*, 1870, **9**, 225), and the reaction is generally represented as:

$$2NH_3 + 3NaOBr = 3NaBr + 3H_2O + N_2,$$

notwithstanding the fact that there is no evolution of nitrogen if ammonia and pure sodium hypobromite are mixed. The evolution of gas only occurs when there is an excess of the sodium hydroxide above that required to form hypobromite. The reaction is probably very similar to the Hofmann reaction (*Ber.*, 1882), in which a brominated compound is formed, which is then decomposed by alkali.

A few qualitative experiments show that the rate of evolution of nitrogen increases with the amount of free sodium hydroxide present, indicating that the rate of decomposition of the brominated compound is dependent on the amount of alkali available for its decomposition.

This reaction can be used for the analysis of ammonium compounds in two ways; either by measuring the amount of nitrogen evolved or by estimating volumetrically the amount of hypobromite unacted upon.

The amount of nitrogen evolved is short of the correct amount, the deficit depending on the nature of the hypobromite solution and the method of collecting the gas. Thus, using a 40 per cent. brominated 10 *N* sodium hydroxide solution, Dietrich (*Zeitsch. anal. chem.*, 1867, **5**, 36) had a deficit which varied from 2 to 3 per cent., and W. Foster (*J. Chem. Soc.*, 1878, **33**, 470) one of 7 per cent., whilst A. Wohl (*Ber.*, 1903, **36**, 1418), using 50 per cent. brominated 1·5 *N* sodium hydroxide solution had a deficit of 4·8 per cent.

The amount of hypobromite which has been unacted upon can be estimated by the method that H. J. Hamburger (*Zeitsch. f. Biol.*, 1885 [3], **20**, 286) used for urea, *i.e.* adding excess of sodium arsenite and titrating this excess by means of a standard solution of iodine in potassium iodide. This method has been tried by D. R. Nanji and W. S. Shaw (ANALYST, 1923, **48**, 473), who found that it gave too high results for ammonium chloride.

Another method of estimating the excess of sodium hypobromite is by acidifying, adding potassium iodide, and titrating the iodine liberated with sodium thiosulphate; this was employed by E. Rupp and E. Rössler (*Arch. Pharm.*, 1905, **243**, 104). They state that for estimating ammonia which contains only a small amount of free alkali it is best to use a hypobromite. The solutions which they used to prove this point were 38 per cent. brominated 2 *N* sodium hydroxide solution and 85 per cent. brominated *N* sodium hydroxide solutions, but the figures that they quote in support are not convincing. H. H. Willard and W. E. Cake (*J. Amer. Chem., Soc.*, 1920, **42**, 2646) used this method to determine the nitrogen converted into ammonia by the Kjeldahl process with accurate results. A very accurate estimation was made by P. Artmann and A. Skrabal (*Zeitsch. anal. Chem.*, 1907, **46**, 5), who obtained good results for various ammonium salts by using 50 per cent. brominated *N* sodium hydroxide solutions.

The reason why the sodium arsenite method gives results which are too high is probably due to reactions which occur in the hypobromite. If bromine is added slowly to a well-cooled solution of sodium hydroxide, the reaction which takes place is as follows:

$$Br_2 + 2NaOH = NaOBr + NaBr + H_2O.$$

If, however, the temperature is allowed to rise, some of the hypobromite is converted into bromate:

$$3NaOBr = NaBrO_3 + 2NaBr.$$

The hypobromite can also break down directly, giving off oxygen gas, especially if traces of a salt of copper, nickel or cobalt are present, but P. Fleury (*Compt. rend.*, 1920, **171**, 957) has shown that this reaction can be inhibited by adding 0·1 per cent. of potassium iodide to the solution.

It has been shown by F. A. Gooch and J. C. Blake (*Amer. J. Sci.*, 1902 (4), **14**, 285) that arsenious acid is only oxidised quickly by bromates in strongly acid, boiling solutions. At ordinary temperatures the reaction requires about an hour for completion, and therefore it is possible that in sodium bicarbonate solution the reaction will not occur at all. Experiments were made by adding sodium bromate solution to sodium arsenite, and then adding sodium bicarbonate, but no reaction could be detected, and the amount of iodine required for a given quantity of sodium arsenite remained constant until the solutions were acidified and heated on the steam bath.

The fact that the sodium arsenite method gives high results may be due to the fact that the reaction between ammonia and hypobromite causes some of the latter to be converted into bromate, and this hypobromite will therefore not appear in the second titration. Gooch and Blake also show that the reaction between acidified bromate and potassium iodide does not quite go to completion, but, as there is very little bromate in the original hypobromite and, also, as two minutes are allowed before titrating with thiosulphate, this error is negligible.

EXPERIMENTAL.

Ammonium chloride.—The ammonium chloride used was Kahlbaum's "Zur Analyse," with guarantee, which had been dried at 105° C.; 1·6 grm. was dissolved in distilled water and the solution made up to one litre, so that 10 c.c. were equivalent to 4·193 mgrms. of nitrogen.

Sodium thiosulphate.—This was standardised by means of potassium dichromate and potassium permanganate and found to be 0·06132 *N*.

Sodium hypobromite.—This was made of a similar strength to that used by Nanji and Shaw by dissolving 2 grms. of sodium hydroxide in two litres of distilled water, which was then cooled in a freezing mixture and 3·4 grms. of bromine slowly added. This procedure ensures that a minimum amount of bromate is formed. The solution was allowed to attain room temperature just before it was required. The strength of this solution was therefore 85 per cent. brominated 0·025 *N* sodium hydroxide solution.

Since it was found that, as the amount of free sodium hydroxide is decreased, the reaction takes more time for completion, the procedure was to add the ammonium chloride solution slowly to the hypobromite in a litre flask (not *vice-versa*) and, after being well shaken, it was allowed to stand for five minutes. The solution was then acidified with hydrochloric acid, and a solution of about 3 grms. of potassium iodide in water added. Sufficient water was added to bring the volume up to 250 c.c., and the iodine was then titrated with thiosulphate after waiting for about 2 minutes to allow the traces of bromate to react completely with the potassium iodide. The results obtained are shown in Table I.

TABLE I.

NaOBr.	Excess.	Thiosulphate, before.	Thiosulphate, after.	Thiosulphate used.
c.c.	Per Cent.	c.c.	c.c.	c.c.
50	9·2	16·09	1·37	14·72
100	119	32·16	17·44	14·74
100	119	32·18	17·42	14·76
150	228	48·24	33·52	14·72
150	228	48·24	33·46	14·78
200	337	64·34	49·62	14·72
200	337	64·34	49·65	14·69
			Mean	14·73

$$14{\cdot}73 \times {\cdot}02044 \times 14{\cdot}008 = 4{\cdot}217 \text{ mgrms. of nitrogen.}$$

As the value obtained was about 0·6 per cent. too high, a fresh estimation was made, the ratio of bromine to sodium hydroxide recommended by Artmann and Skrabal (*i.e.* 50 per cent. brominated 0·025 *N* sodium hydroxide solution) being used. These results are given in Table II.

TABLE II.

NaOBr. c.c.	Excess. Per Cent.	Thiosulphate, before. c.c.	Thiosulphate, after. c.c.	Thiosulphate used. c.c.
50	—	9·42	—	—
100	28·5	18·87	4·19	14·68
100	28·5	18·83	4·18	14·65
150	92·6	28·26	13·60	14·66
150	92·6	28·26	13·60	14·66
			Mean	14·66

14·66 × ·02044 × 14·008 = 4·198 mgrms. of nitrogen, or an error of about 0·1 per cent.

These results show that, providing the procedure detailed above is followed, the estimation of ammonia by means of sodium hypobromite is a very accurate method.

In order to compare this method with the arsenious oxide method of Nanji and Shaw experiments were made, with the following results:

Thiosulphate = 0·06132 *N*.
20·00 c.c. iodine in KI = 12·81, 12·81, mean: 12·81 c.c. thiosulphate.
5 c.c. sodium arsenite = 12·65, 12·64, mean: 12·65 c.c. iodine.
25 c.c. NaOBr + 10 c.c. As_2O_3 = 11·42, 11·42, mean: 11·42 c.c. iodine.
25 c.c. NaOBr + HCl + KI = 8·96, 8·95, Mean: 8·96 c.c. thiosulphate.
Hypobromite by arsenious oxide method = 0·02180 *N*.
Hypobromite by thiosulphate method = 0·02197 *N*.

50 c.c. NaOBr + 10 c.c. NH_4Cl + 5 c.c. As_2O_3 = 6·24, 6·50, 6·74, 7·16, 7·06 c.c. iodine, corresponding to 3·91 to 4·08 mgrms. of nitrogen and about 22 per cent. excess of hypobromite.

The ammonium chloride was checked against some twice re-crystallised ammonium chloride from another source, and the two gave identical results when acidified and titrated with thiosulphate after adding potassium iodide.

SUMMARY.—(1) The most accurate method of estimating ammonium salts by means of sodium hypobromite is a modification of that used by Artmann and Skrabal.

(2) Small quantities of ammonia corresponding to 4 mgrms. of nitrogen can be estimated by means of 50 per cent. brominated sodium hypobromite solution with an accuracy of about 0·1 per cent.

DEPARTMENT OF PHYSICAL CHEMISTRY,
IMPERIAL COLLEGE, S.W. 7.

Note.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

A QUALITATIVE DIFFERENTIATION BETWEEN CUBE GAMBIER AND INDIAN CUTCH.

IT has frequently been noticed in this laboratory that whereas cube gambier gives a green-coloured extract on boiling with chloroform, no such coloration is obtained when authentic Indian cutches are treated in the same manner.

Since cube gambier is prepared from the leaves of *Uncaria Gambier*, whereas the Indian cutches are prepared from the heart-wood of *Acacia catechu*, *A. catechuoides* and *A. sundra*, it seemed possible that the green coloration of the chloroform extract was due to chlorophyll. I have, at the suggestion of Dr. Nierenstein, investigated cube gambier, and have found that the chloroform extract contains chlorophyll.

About 1 grm. of cube gambier was heated for a short time on a boiling water bath with 10 c.c. of chloroform, and the filtered solution examined in a direct vision spectroscope, when a distinct spectrum, corresponding to that of chlorophyll, was obtained.

The test is fairly delicate, as will be seen from the following experiments:

Cutch. grm.	Chloroform. c.c.	Colour of Extract.	Remarks.
2	7	dark green	Definite chlorophyll spectrum.
1	7	dark green	Definite chlorophyll spectrum.
0·5	7	pale green	Faint spectrum.
0·3	7	pale green	Chlorophyll not detectable.
0·2	7	yellowish	Chlorophyll not detectable.

From the results of these experiments I would suggest using 1 to 2 grms. of cube gambier and 7 to 10 c.c. of chloroform for the detection of chlorophyll in cube gambier. In this connection it must be mentioned that one of the cube gambier specimens examined gave a good chlorophyll spectrum when only 0·3 grm. of it was extracted with 7 c.c. of chloroform.

None of the Indian cutches examined answered to this test, which can therefore be used as a method for the differentiation of cube gambier from Indian cutches. This is of importance, as it is known that adulterated gambier cutches are placed on the Deccan markets as genuine Indian cutches. (*Cf.* D. Hooper, *Indian Agricultural Ledger*, **1906**, p. 44.)

R. P. BIGGS.

BIOCHEMICAL LABORATORY,
UNIVERSITY OF BRISTOL.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE 1ST QUARTER, 1924.

DURING the quarter, 1456 samples were examined, of which 1264 were submitted under the Sale of Foods and Drugs Acts, 1077 being bought informally (of which 51 were adulterated), and 187 bought under the provisions of the Acts (of which 20 were adulterated).

MILK.—Of the 669 samples examined, 45 were reported as adulterated.

CONDENSED MILK.—Only one of 4 informal samples of full cream sweetened condensed milk was satisfactory. One contained 8·7 per cent., and a second 8·8 per cent. of fat, instead of the minimum 9 per cent., whilst the directions for diluting the third sample did not appear to comply with the regulations. In each case the manufacturer was cautioned.

MUSTARD.—Sixteen samples were genuine, but two, bought at one shop, contained about 28 per cent. of potato starch and turmeric.

GROUND GINGER.—Ten samples were genuine, and 4 adulterated. Two of these (a formal and an informal sample from the same vendor) contained about 2 per cent. of chalk. Two other samples (formal and informal) from the same vendor were deficient in soluble pungent principles. The formal sample contained 3·0 per cent. of ash, of which 1·80 per cent. was soluble in water, and 0·34 per cent. insoluble in acid. The cold water extract was 6·6 per cent. The average composition of 250 samples of ground ginger purchased in Birmingham during the past 30 years was as follows:—Ash, 5·34 per cent., of which 2·54 per cent. was soluble in water, and 0·63 per cent. insoluble in acid. Cold water extract 12·7 per cent. The two owners of the shop were each fined 5s.

CUSTARDS.—Nine samples were free from boric acid. Three, examined in detail, consisted on the average of 36 per cent. of custard and 64 per cent. of pastry. The average percentage composition was as follows:

	Custard.	Pastry.	Total.
Protein	8·4	5·6	6·6
Fat	8·3	16·9	13·8
Ash	1·0	0·4	0·6
Moisture	67·9	35·2	47·0
Starch, etc.	14·4	41·9	32·0
Nitrogen	1·29	0·87	1·01

ALE.—One informal sample and one formal sample from one vendor contained chlorides equivalent to about 100 grains of salt per gallon, and the vendor was cautioned.

PURE SULPHATE OF IRON.—The pure chemical contains 45·4 per cent. of moisture, some of which is lost on exposure to the air, so that the drug becomes stronger. The B.P. requires the salt to be crystalline and of a greenish colour. Before 5 per cent. of moisture is lost the drug becomes white and powdery, and would not comply with the B.P. requirements. Six informal samples contained the equivalent of 100·3 to 101·5 per cent. of pure ferrous sulphate, and were passed as genuine. About 1 oz. of the granular sulphate was kept in an open bottle for 28 days, and during that period lost 1·9 per cent. of moisture. When freely exposed to the air, 9 samples lost from 18·0 to 20·4 per cent. of moisture in about 12 days and showed no further loss. This amount corresponds to about 3/7 of the water of crystallisation.

QUININE SULPHATE.—None of the six informal samples contained 15·2 per cent. of water—the amount implied in the composition of the salt as stated in the B.P.—the percentage varying from 4·6 to 12·0. It is suggested that the statement in the B.P., that quinine sulphate loses water of crystallisation, is not intended to justify the use of such an indefinite substance. It is probable that this tendency of quinine sulphate to become stronger through loss of water will account for the fact that dispensed mixtures containing quinine sulphate have been found too strong, and also for the excess of quinine found in B.P. preparations, such as ammoniated tincture of quinine.

J. F. LIVERSEEGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

COMPOUND CASTOR OIL TABLETS.

ON April 14th a druggist was charged at the Magistrate's Court, Wanganui, New Zealand, with selling a drug in a package, to wit, compound castor oil chocolates, bearing and having attached thereto a false or misleading statement. The analyst's report stated that "the material was in a form of chocolate coloured tablets of an average weight of 1·55 grms., and contained matter soluble in petroleum spirit 4·4 per cent., phenolphthalein, 7 per cent. Not more than half the matter soluble in petroleum spirit was castor oil, and it was possible that no castor oil was present. Assuming that the material contained 2·2 per cent. of castor oil, 116 tablets would be required to provide a minimum dose of that substance. The dose prescribed for a child of three to ten years was one tablet. It was evident, therefore, that the quantity of castor oil present would not have the slightest medicinal action. The active ingredient was the phenolphthalein. The statement "Compound Castor Oil Tablets" appearing on the original package was misleading, and in contravention of Sec. 12 of the Sale of Food and Drugs Act, 1908."

Counsel for the Department of Health pointed out that the ordinary dose of phenolphthalein was 2 to 5 grains, whilst the amount in the tablets was 1·8 grains. The point was, he contended, that the tablets did not contain castor oil, when the label said they did. There might be an aperient of a certain nature, but this was not material to the case.

Counsel for the defence said that the tablets were purchased from a well-known manufacturer in London. The chocolates were not stated to be castor oil chocolates, but compounded castor oil chocolates, and this statement showed that there was something else present besides castor oil. Assuming each tablet to contain 2 per cent. of castor oil and 7 per cent. of phenolphthalein, castor oil would constitute 25 per cent. of the aperient agent. He contended that there was no deception of the public, as phenolphthalein was more expensive than castor oil.

The Magistrate observed that, from the point of view of the man in the street, he would think from seeing the words "Compound castor oil chocolate" that the effective chemical agent was castor oil, and for that reason the statement on the label was misleading. The defendant could have protected himself by warranty or by analysis. He found that there had been an infringement of the Act, but did not think the case was one for severe penalty. A fine of £1 and costs was imposed.

FISH MEAL WITH EXCESSIVE OIL.

On June 4th, a feeding-stuffs merchant was charged at Newport Pagnell Petty Sessions with selling fish meal under an invoice which did not accurately set out the contents of the article sold, and contrary to the provisions of the Fertilisers and Feeding Stuffs Act, 1906.

On February 7th, 2 tons of fish meal were ordered from the defendant, who, on the same day, sent an invoice to the purchaser, which gave the following analysis of the meal: Oil, 4½ per cent.; albuminoids, 56 per cent.; salt, under 3 per cent.

The meal was delivered on February 8th, and on February 15th, at the request of the purchaser, it was sampled by the Chief Inspector for the County.

Samples were submitted to Dr. J. A. Voelcker, the Agricultural Analyst for the County, who reported that the meal had the following composition: Oil, 11·88 per cent.; albuminoids, 50 per cent.; salt, 2·34 per cent.; and sand, 2·29 per cent. The Analyst added to his report the comment: "This is a very fluffy sample. Oil is much—and objectionably so—in excess of what is stated in the guarantee. Sand is somewhat high. Albuminoids are somewhat low, doubtless on account of the excessive oil."

This report was submitted to the Ministry of Agriculture and Fisheries, where it was decided that a prosecution should be instituted.

Dr. J. A. Voelcker, giving evidence in support of his certificate, said that the excess of oil, after making the usual statutory and other allowances, was about 6½ per cent., an excess which was open to very strong objection on two grounds. One was the liability of the meal to upset the animal eating it. The other was a known tendency for fish oil to make bacon fat taste. Excess of fish oil had a prejudicial effect. That was so much recognised by the trade themselves that the Association of Manufacturers of Fish Meal had agreed amongst themselves to give a guarantee that their fish meal should not contain more than a certain percentage of oil. That percentage was 4½ as a rule, or 5 per cent. If it went materially above 5 per cent. he would consider the sale to be to the prejudice of the purchaser. Albuminoids were valuable food constituents, and in this sample he found 50 per cent., instead of 56 as stated on the invoice.

Mr. James G. Stewart, Deputy Chief Inspector to the Ministry of Agriculture, and technical adviser on agriculture, stated that fish meal containing 11·88 of oil was certainly to the detriment of the purchaser on account of the liability of such a high percentage to cause taint in flesh, such as bacon; also in eggs and poultry.

Albuminoids were highly valuable in food value; therefore a deficiency of 6 per cent. was to the detriment of the purchaser, and a sample containing over 2 per cent. of sand was harmful, sand being entirely indigestible. The present sample, according to its analysis, was more suited to use for fertilising than feeding purposes.

The defendant stated that the fish meal in question was sent direct from the manufacturers. He had based his invoices on the analysis obtained from them, and he was honestly of opinion that those invoices were correct within the prescribed limits.

In cross-examination, he said that, notwithstanding a complaint he had received about a former consignment, he had not himself taken steps to have the meal analysed. The risk was the manufacturer's, not his.

Counsel for the defence submitted that the defendant was the innocent victim of circumstances, in the capacity of agent, who never saw the fish meal.

The prosecution, on the other hand, contended that the defendant had not taken any reasonable care to satisfy himself that the invoice was correct, even in the face of a complaint made to him by the same purchaser, in December, in respect of another consignment.

The Magistrates decided that reasonable care was not taken by the defendant, and they imposed a fine of £20, with £10 10s. costs.

Report of the Departmental Committee on the Fertilisers and Feeding Stuffs Act, 1906.*

In July, 1923, a Departmental Committee was appointed by the Minister of Agriculture and Fisheries to enquire into the operation of the Fertilisers and Feeding Stuffs Act, 1906, to advise whether any, and, if so, what, amendments are necessary in order to render the execution of the Act more economical and effective, and to report accordingly.

The following members were appointed:—The Lord Clinton (Chairman), Messrs. E. R. Bolton, E. G. Haygarth Brown, C. Crowther, T. Kyle, B. S. Miller, G. Stubbs, J. F. Tocher, and J. A. Voelcker.

Evidence was received from nineteen witnesses, representing agricultural societies, merchants, manufacturers, importers, agricultural analysts, and County Councils' Associations, and this evidence is summarised in the Report.

In the introduction (Part I.) it is pointed out that many of the questions which would otherwise have needed close consideration, have already been exhaustively dealt with in the Reports of Committees set up previously.

With regard to questions of detail in connection with the specific constituents of fertilisers and feeding stuffs which would be included in a warranty, the Committee is of opinion that these would be more effectively dealt with by an *ad hoc* Committee.

A Memorandum prepared by the Ministry was circulated to all those called upon to give evidence, and it has been ascertained that, in every case, this has been accepted as a general basis on which new legislation may be founded.

In some of the recommendations the Committee has been influenced by the fact that these agreements point the way to an even balance being obtained between the conflicting views of rival interests.

* H.M. Stationery Office, Cmd. 2125. Price 1s. net.

The Report gives a historical note on the Acts of 1893 and 1906, and on the Sales for Agricultural Purposes Bill.

In Part II. it discusses the principal objections to the present Act under the following headings:—(1) The Position of the Farmer; (2) The Claims of the Merchant and Manufacturer; (3) The Case of the Importer; (4) Difficulties in Administration.

Part III. discusses proposals to remove the objections to the present Act, under the following headings:—(1) Civil Provisions; (2) Criminal Provisions; (3) Exceptional Provisions for Sales in Small Quantities; (4) Local Authorities entrusted with Enforcement of the Act; (5) Definition of the Scope of the Act; (6) Protection of the Seller against unmerited Prosecution.

Part IV. gives a summary of the principal recommendations. These would make so great a change in the existing law that they cannot be met by merely amending the Act of 1906. These recommendations are as follows:

(I) The bases of civil and criminal procedure should be clearly separated, the invoice remaining the foundation of civil claims and the description applied to the goods becoming that of criminal proceedings.

(II) The statement in the invoice should be a warranty, as at present, and no statement in the invoice should be held to relieve the seller of his civil liability.

(III) The period allowed for sampling should be extended to fourteen days after delivery of the goods or receipt of the invoice, and the requirement that notice of sampling be given to the seller should be abolished.

(IV) No sample other than those defined on page 24 as "formal" or "informal" should be analysed at the expense of the rates.

(V) The definitions in the Schedules suggested in sub-paragraph (XX) below should have effect as a warranty.

(VI) No criminal prosecution for the application of a false description should take place except in respect of a sample taken on the premises of the person to be prosecuted, or in the course of transit in a vehicle into which the consignment is loaded by him or his servants, or in the first conveyance of a public carrier into which it is loaded.

(VII) In the case of samples taken in transit, it should be necessary to provide conclusive proof that they have been taken in accordance with the Act and Regulations.

(VIII) Failure to give statutory invoice should be an offence under the Act in respect of which a prosecution may be commenced by any Local Authority administering the Act, and without the consent of the Government Department concerned. It should not be necessary to take and analyse a sample before prosecuting for this type of offence.

(IX) Every parcel of goods to which the criminal provisions of the Act apply should, when prepared for consignment or delivery, be marked in such a way as will indicate to the purchaser the name and address of the consignor and the description of the goods. It should be permissible for the name and address of the consignor to be applied by means of a mark registered with the Government Department concerned, and for the description to take the form of a code sign, of which the index must be displayed for inspection on the premises of the consignor, and a copy deposited with the Ministry or the Board. Failure to apply the description (or code sign) and the name and address of the consignor (or registered mark), or either of them, before consignment should, save in such cases as those referred to on pages 30 and 36, be an offence under the Act.

(X) A power of entry into all ports, factories, warehouses, stores and shops in which fertilisers or feeding stuffs may be manufactured or stored should be given to authorised inspectors, together with a power to take samples of goods prepared for consignment or delivery, and also a similar power of entry and taking informal samples of any article on the premises when it appears that the analysis of such article may throw light on the question whether fraudulent adulteration has, or has not, taken place on those premises. A power of entry to, and taking samples on, all farms should also be given.

(XI) Applying a false description to goods prepared for consignment should be an offence under the Act.

(XII) The presence of a deleterious substance in a feeding stuff, or the addition to a feeding stuff of a worthless ingredient the presence of which is not disclosed, should be an offence under the Act. For this purpose, Schedules to the proposed Act should be prepared to show those substances, with regard to which there may be reasonable doubt, which are to be regarded as "worthless" or "deleterious" for this purpose.

(XIII) Any discrepancy to the prejudice of the purchaser between the description attached to a parcel of goods and the invoice given in respect of it should constitute an offence under the Act.

(XIV) Where an article is described by a name comprising a name defined in the Schedules, with or without qualifying words, it should be implied that the article consists of that substance only.

(XV) Prosecutions for the offences set out in paragraphs (IX), (XI), (XII) and (XIII) should not be instituted by any Local Authority except with the consent of the central Government Department.

(XVI) No invoice should be required to be given with quantities of goods of not more than half a hundredweight sold from bulk, provided the prescribed particulars are clearly stated on a label attached to the bulk and plainly visible to the purchaser, nor with packeted goods of not more than half a hundredweight if the description appears on the package.

(XVII) The duty of administering the Act should be imposed by Statute on all County Councils and County Borough Councils in England and Wales, and all County Councils and Burgh Councils in Scotland. Power should be given to Local Authorities to establish joint Committees for the purpose of carrying out the Act, and also to exercise their powers of entry, sampling and prosecution in the area of any other Local Authority with the consent of that Authority. Concurrent powers of entry, sampling and prosecution should be vested in the Ministry of Agriculture and Fisheries and the Board of Agriculture for Scotland.

(XVIII) Local Authorities should be required to make public, at least half-yearly, the results of analyses of all formal samples, together with the name and address of the seller or consignor and the warranty given in each case.

(XIX) Inspectors under the Act should be senior wholetime officials of the Local Authority. It should be open to the Local Authorities to appoint, in addition to these Inspectors, other official Samplers for the purpose of taking samples.

(XX) The scope of the Act should be defined by Schedules which should indicate, in respect of fertilisers and feeding stuffs, (*a*) the name of each article or class of article to which the Act should apply; (*b*) the definition of the substance named; (*c*) the particulars to be stated in the invoice for the purpose of the civil warranty; and (*d*) the particulars to be stated in the description for the purpose of the criminal provisions of the Act. Provision should be made for amendment of these Schedules, and also those mentioned in paragraph (XII), by Regulations made by the Minister and the Board of Agriculture for Scotland, acting jointly.

(XXI) The defences set out in Section 2 (2) of the Merchandise Marks Act, 1887, should be provided in place of those afforded by the present Act.

(XXII) It should be provided that no civil proceedings, and no criminal proceedings in respect of the application of a false description, shall be instituted in the case of any sample unless the results of the analysis of the sample differ from the particulars stated in the invoice or in the description, as the case may be, by more than the amounts prescribed in the "limits of variation."

(XXIII) An Advisory Committee, which should subsequently become a statutory Standing Advisory Committee, should be constituted for the purpose of discharging the following functions:

1. To draw up Schedules for the purpose of prescribing:
    (*a*) The fertilisers and feeding stuffs to which all the provisions of the Act should apply and those to which only the civil provisions should apply;
    (*b*) Definitions of each of the articles or classes of articles to which the Act should apply;
    (*c*) The statements as to the constituents present and also, possibly, as to the absence of certain substances in some instances, which should be given by the seller for the information of the farmer in the case of each article;
    (*d*) Those commodities which should be regarded as "worthless" or "deleterious."
2. To recommend the terms in which the valuable constituents should be stated in the invoice (*e.g.* whether phosphates should be stated as phosphoric acid or as tri-calcium phosphate).
3. To review the limits of variation and recommend any necessary alterations or additions.
4. To consider the form of code signs and registered marks and the methods of application.
5. To review the existing Regulations and suggest such amendments and additons as may be thought desirable.
6. To recommend, from time to time, such alterations to the Schedules and Regulations as may become necessary.

In Part V. changes less fundamental in character are recommended. These refer to Sections of the Act of 1906 which, in the opinion of the Committee, contain provisions that will still be of value. The amendments suggested are as follows:

Section 1, *subsections* (1) *and* (2) appear to require re-drafting in accordance with our main recommendations.

Subsections (3), (4), (5) *and* (6) might usefully be incorporated, in principle, in fresh legislation.

Section 2, *subsection* (1) should be replaced by a provision that the Chief Agricultural Analyst shall be the Government Chemist.

Subsection (2) will require amendment, in accordance with our proposal to impose a statutory duty on all County Councils and Councils of County Boroughs to enforce the Act.

Subsection (3) seems to need no alteration.

Subsection (4) should, we think, be amended, as regards the appointment of Inspectors, in accordance with the recommendations made on page 39 of this Report.

Subsection (5). This provision should be retained and made applicable to Inspectors and Official Samplers as well as to Analysts and Deputy Analysts; but it should be provided that a person who sells agricultural produce which he has himself grown should not thereby be debarred from holding any of these appointments.

Section 3, *subsections* (1) *to* (5) *and* (7) require amendments consequential to our main recommendations.

Subsection (4) (b) should, further, be amended by the addition of the words "or cause to be analysed under his direction" after the word "analyse." This subsection should also be altered, in our opinion, so as to provide that a copy of the Agricultural Analyst's certificate shall be sent to the seller only where the results of the analysis differ from the warranty to the prejudice of the purchaser and by more than the amounts prescribed in the limits of variation.

Subsection (6) should, we think, be amended so as to provide for an analysis of the reserved part of a formal sample being made by the Chief Analyst, for the purpose of civil proceedings, at the request of either party and on payment of the required fee, either before or after the commencement of the proceedings.

Section 4. The proposals we have made involve wider powers to make Regulations, and the Section would, therefore, need consequential amendments.

The proviso to subsection (1) should, in accordance with paragraph (IV) of the Summary of Principal Recommendations, be deleted.

Section 5. The principal alteration we have suggested which would affect this Section is that Local Authorities should be empowered to set up Joint Committees for the purpose of carrying out the Act.

It has also been suggested that Councils should be empowered to make purchases, and we consider it necessary that this power should be specifically granted.

Section 6. The whole of this Section, with the exception of subsections (1) (*a*), (2) and (4), would seem to require complete redrafting.

Subsection (3) should be framed so as to permit of the analysis of the part of the sample retained by the Agricultural Analyst either by the Chief Analyst or under his direction.

Section 7 should, we think, be reproduced without alteration.

Section 8, we suggest, should remain in force and be extended by the addition of the words "or hinders, obstructs or prevents his taking such sample, or is a party to such hindrance, obstruction or prevention" after the words "purpose of analysis" at the end of paragraph (*a*).

Section 9 would seem to require merely drafting amendments in order to bring it into concert with the general suggestions we offer.

The provision made in subsection (2) of this Section as to the place of hearing of certain proceedings should apply to all cases of prosecution under the Act.

Section 10. We have already discussed the question of the method of stating the percentage of valuable constituents, and recommended that this matter should be dealt with by the Advisory Committee.

As regards the meaning of terms, we suggest that "delivery" should be defined so as to provide that it shall mean arrival of any consignment, whether part or whole of the purchase, at the purchaser's station, wharf or premises, or the earlier loading by him on to his own vehicle.

Ministry of Health.

REPORT ON THE COMPOSITION OF COMMONER BRITISH WINES AND CORDIALS.*

THIS report is based on a special inquiry begun in 1913, interrupted by the war, and only recently completed. It comprises information supplied by the manufacturers of British Wines and Cordials, and a classified summary of the results of the analysis of 357 samples by the Government Chemist.

SALE AND DISTRIBUTION.—Although, strictly speaking, the term "wine" should be restricted to the fermented expressed juice of any fruit, the term "British wine" has been applied to a number of beverages prepared in the British Isles, some of which contain alcohol produced, in part, by the fermentation of fresh and dried fruits, whilst others consist, for the most part, of sugar solutions flavoured with fruit juices and essences, and contain no alcohol, or only an amount not exceeding 2 per cent. of proof spirit, to which exception is not taken by the Excise Authorities. These are almost invariably described as "non-alcoholic" or "unfermented."

The annual output of fermented wines probably does not exceed 2 million gallons per annum, but the output of temperance wines and cordials cannot be estimated even approximately.

Although wine from British-grown grapes is no longer an article of commerce, a small quantity is manufactured from imported foreign grapes by fermenting them with added saccharine matter, and a very considerable quantity is made from imported grape must.

FERMENTED BRITISH WINES.—A small amount of wine is made by fermenting the juice of various fruits, with the addition of sugar, but by far the larger proportion consists of a more or less characterless wine, made by fermenting an infusion of raisins, dried currants or rhubarb, or imported grape must, with or without added sugar, and adding distinctive essences or flavouring agents (ginger, orange peel, fruit extracts). At one time a large proportion of foreign wine was frequently added, but recent excise regulations prohibit the addition of more than 15 gallons per pipe of foreign wine to British wines

After fermentation, the wines are fined with gelatin or isinglass or by means of siliceous earths, such as Spanish clay. Colouring is effected by the addition of caramel, dried elderberries, or other vegetable colours, and, exceptionally, by aniline dyes. Of 145 alcoholic wines examined by the Government chemist, only 7 contained aniline dyes. The preservatives most commonly used are sulphurous acid and sulphites, used either in the form of burning sulphur or as bisulphite. Nearly all the samples examined contained some sulphurous acid; 14 per cent. contained it in excess of 200 mgrms. per litre; and 18·6 per cent. contained between 100 and 200 mgrms. per litre. Fourteen of 134 samples contained salicylic acid (0·25 to 1·57 grains per pint), and fluorides in small quantity were present in 8 samples. Seven samples contained boric acid in amounts ranging from 1·38 to 2·98 grains per pint. Many samples contained traces of boric acid, which has been shown to be present in the ash of genuine wine.

The proof spirit in basis wines was found to range from 14·8 to 28 per cent., and in ginger wines from 10·9 to 27 per cent. Most of the samples contained sugar to such an extent as to give them the character of sweet wines, and, in a few cases, "cordials."

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* Reports on Public Health and Medical Subjects, No. 24, by G. C. Hancock, G.B.E., M.R.C.S., L.R.C.P., together with Report by the Government Chemist on the Examination of Samples. H.M. Stationery Office. Price 1s. 6d. net.

BRITISH UNFERMENTED WINES AND CORDIALS.—These usually consist of a basis of syrup made from cane or beet sugar, supplemented, in some cases, by saccharin and, more rarely, by glucose. Of 44 non-alcoholic wines examined by the Government Chemist, saccharin was found in 10, and of 98 cordials, it was found in 7. The amount ranged from traces to 4·6 grains per pint.

The syrups are flavoured with fruit extracts, fruit essences, and artificial flavouring substances, and usually contain (1) vegetable acids, (2) colouring matters, (3) preservatives.

Of the vegetable acids, citric and tartaric acid are most commonly used. A phosphoric acid preparation called "phospho-citric acid" is sometimes used as a substitute.

Caramel and vegetable colouring matters are frequently used, and coal-tar dyes are now widely employed. Over 30 per cent. of the samples examined contained aniline dyes. These are referred to in detail in Part II. of the Report.

Preservatives.—Of 142 samples examined, 71 contained salicylic acid in amounts ranging from a trace to 11·9 grains per pint. Sulphurous acid was present in traces in nearly all the samples. In 29 cases the amount exceeded 100 mgrms. per litre. Boric acid was found in 18 samples, in amounts ranging from 1·01 to 14·31 grains per pint, and, in all but 4 cases, it was present in addition to salicylic acid or sulphurous acid.

Benzoic acid was found in 2 samples (4·7 and 10·6 grains per pint), and small quantities of fluoride were detected in 2 other samples.

As a rule, non-alcoholic wines do not contain more than 2 per cent. of proof spirit, but two samples from the same firm were found to contain 23 per cent.

Metals.—Twenty-four of the alcoholic wines and 19 of the non-alcoholic wines and cordials were examined for lead, copper and tin. None contained lead or tin, but copper was found in 22 samples in quantities ranging from 0·01 to 0·10 grain per pint. In view of this fact, the Government Chemist points out that copper has been found to occur naturally in a large number of cereals, fruits and fruit juices. (A list of references to the literature on the subject is given.) Arsenic was not found in any of the samples examined.

Tables giving the analytical details obtained with each of the samples occupy the concluding 26 pages of Part II. of the Report.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Microscopic Detection of some Fortified Milks. D. W. Horn. (*Amer. J. Pharm.*, 1924, **96**, 365–366.)—The microscopic appearance of the centrifugal residues from milks is sufficiently characteristic for the ready detection of 1 part of condensed milk products in 60 of milk. The milks are centrifuged for 10 minutes at 2000 to 2100 revolutions per minute in glass tubes closed at the bottom with rubber stoppers. Smears from the residue adhering to the stoppers are air-dried and stained with methylene blue (1·25 grm. of dye in 60 c.c. of wood alcohol.) In the case of fortified milks microscopic examination reveals numerous foreign bodies suggestive of minute starch grains. Unlike fat globules, these stain, and sometimes have the appearance of collapsed spheres. Possibly they are colloidal gel particles, as any non-reversible colloids present in the milk would, presumably, be deposited on the application of sufficient centrifugal force. The smears from fortified milks usually appear to the naked eye as lustreless and rough, with matte surfaces. D. G. H.

"Kaschkawal." A Bulgarian Cheese. A. Heiduschka. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 260–261.)—Kaschkawal, a Bulgarian cheese made from sheep's milk, or occasionally from goat's milk, is now being exported in large quantities. It appears as a pale yellow cheese similar to Gruyère and, like it, has air-spaces of 0·5 to 1 cm. diameter. The mean result of 17 analyses was as follows:—Water, 29·0; fat, 29·0; proteins, 31·3; and ash, 6·8 per cent.; refractometer number of the fat at 40° C. 41·5. Details are given of the cultural characteristics of the typical bacteria. H. E. C.

Polarimetric Estimation of Starch in Potted Meats. A. During. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 248–251.)—The following simple polarimetric method for the estimation of starch is shown to give accurate results, which also compare well with the well-known Mayrhofer method. Ten grms. of the meat are boiled on the water bath under a reflux condenser with 25 c.c. of an 8 per cent. alcoholic solution of potassium hydroxide; then 25 c.c. of hot 5 per cent. alcohol are added, and the mixture is filtered on to asbestos with the aid of the pump; in the case of very fatty foods a second treatment of the starch with alcoholic potash is desirable. The filter and the starch are mixed with a small volume of water, made neutral to phenolphthalein, after which 25 c.c. of diluted hydrochloric acid (40 c.c. of acid of sp. gr. 1·124 diluted to 1 litre) are added, and the mixture is heated in boiling water for 15 minutes; it is then cooled and made up to, say, 50 c.c. The rotation is observed at 20° C. in a 200 mm. tube. Working on 10 grms. of material and making the final volume 50 c.c., the starch is calculated from the equation:

$$\frac{\text{Observed rotation} \times 1{\cdot}775 \times 2{\cdot}88}{4} = \text{per cent. of pure starch;}$$

$$\times \frac{5}{4} = \text{per cent. of commercial starch.}$$

H. E. C.

New Method of Distinguishing between Honey and Artificial Honey. F. Auerbach and E. Bodländer. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 233–238.)—The essential difference between genuine and artificial honey lies in the ratio of fructose to glucose; in the former this amounts to 106–119:100, and does not increase on heating or by prolonged storage. In artificial honey the ratio is less than 90:100. For the analysis the honey is warmed to about 50° C. to obtain a clear homogeneous mixture; then 2 grms. are weighed out, diluted to 250 c.c., and the total reducing sugar is estimated gravimetrically on

25 c.c. The glucose is estimated iodimetrically by adding a large excess of 0·1 *N* iodine solution, which should be about twice the iodine used, to 25 c.c. of the honey solution, together with 100 c.c. of a mixture of equal parts of 0·2 *M* sodium bicarbonate and carbonate solutions, the mixture being allowed to stand in the dark for 1½–2 hours. (*Cf.* ANALYST, 1923, **48**, 73.) The liquid is then acidified with 12 c.c. of 25 per cent. sulphuric acid and titrated with 0·1 *N* thiosulphate solution. A blank titration is made at the same time, with the use of similar quantities of the iodine, carbonate and bicarbonate, and sulphuric acid solutions. The difference between the two titrations represents the glucose: 1 c.c. 0·1 *N* iodine = 0·009005 grm. of glucose. The difference between the glucose and total reducing sugars is reckoned as fructose; then the above ratio is calculated.

H. E. C.

Iron Content of Spinach. A. Lichtin. (*Amer. J. Pharm.*, 1924, **96**, 361–364.)—The percentage of iron in different varieties of spinach was found to vary between a maximum of 0·0087 for Long Standing and a minimum of 0·0027 for New Savoy, with an average of 0·0051 for truck spinach, 0·0568 for dehydrated spinach, and 0·00076 for canned spinach. The estimations were made by comparing the colours produced in definite concentrations of the acid extractions of the ash with standard iron solutions prepared by oxidising the ferrous salt formed from ferric ammonium sulphate and sulphuric acid with 0·1 *N* potassium permanganate solution. Most of the results are compared with those obtained by titration with potassium permanganate solution. D. G. H.

Cacao Germ. F. Härtel. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 264–266.)—Beythien and Pannwitz's observations on cacao germ (*ibid.*, 1923, **46**, 230) are considered defective because they included some of the kernel; a carefully separated sample of germ gave the following analytical results: Water, 2·21; fat, 3·85; nitrogen, 5·25; sucrose, 2·98; ash, 6·76; insoluble ash, 4·46 per cent. The fat had a refractometer reading, at 40° C., of 62·5, iodine value 68·8, saponification value 169·8. The high refractometer number and high iodine value is a characteristic feature which is useful for detecting the presence of the germ in cacao products. The bye-product known as "Kakaogrus" consists mainly of ground kernels and contains a higher proportion of fat than does the germ. The fats in the kernel and shell have much lower refractometer numbers, namely, 47·6–49·1 and 50–53·5 respectively. H. E. C.

Estimation of Dulcin by means of Xanthydrol. G. Reif. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 238–248.)—In 50 per cent. acetic acid solution at ordinary temperatures dulcin combines quantitatively with xanthydrol, forming exactly twice its weight of xanthyl-dulcin, which is a colourless crystalline substance of m.p. 246° C., and is readily soluble in water, alcohol, ether and similar solvents. This reaction is useful for the estimation of quite small quantities of dulcin. As saccharin and certain other substances are liable to interfere, they are eliminated by lead acetate precipitation and subsequent extraction of the dulcin with ethyl acetate in alkaline solution. The method, as applied to a fruit syrup, is as follows:—Fifty grms. of the syrup, diluted with 60 c.c. of water, are acidified with 20 drops of acetic acid, 10 c.c. of a 20 per cent. solution of lead acetate solution are added, and the mixture is allowed to stand for an hour. Then, after ascertaining that excess of lead has been added, the precipitate is filtered off, and the lead removed from the filtrate by the addition of 20 c.c. of a saturated solution containing 20 grms. of sodium sulphate and sodium phosphate in 100 c.c. After allowing the mixture to stand for a few hours it is filtered, and the filtrate made just alkaline and then shaken out twice with an equal volume of ethyl acetate, which is the best solvent for extracting dulcin. The mixed ethyl acetate extract is distilled on a water bath, and the residue is dissolved in 15 c.c. of boiling water and filtered; then the aqueous solution is evaporated to dryness on the water bath, and the residue of crude dulcin is dissolved in 20 c.c. of 50 per cent. acetic acid, the solution filtered, and the filter washed with a further 20 c.c. of the 50 per cent. acid. To this solution is added 0·2 grm. of xanthydrol dissolved in 3 c.c. of hot methyl alcohol; after standing for about 2 hours the precipitate is filtered off on to a Gooch crucible, washed with a small quantity of the acetic acid, dried at 100° C., and weighed.

H. E. C.

Ethylene Glycol. Its Properties and Uses. H. C. Fuller. (*Ind. Eng. Chem.*, 1924, **16**, 624–626.)—Ethylene glycol, $C_2H_4(OH)_2$, sp. gr. 1·116, is a colourless, odourless liquid having a sweetish taste; it is miscible with water in all proportions and bears a considerable resemblance to glycerol, although less viscous than this substance. It appears to have some use as a solvent, particularly in the preparation of flavouring extracts and pharmaceutical products, in which it may replace alcohol. As a preservative ethylene glycol is about equal to alcohol, and is much superior to glycerol; when taken in moderate doses it has no injurious physiological action.

W. P. S.

Analysis of Pharmaceutical and Bacteriological Gelatins. E. Cattelain. (*J. Pharm. Chim.*, 1924, **29**, 444–447.)—Certain modifications of the French Pharmacopoeia requirements are suggested. Since all the samples examined were found to be acid and not neutral, the *acidity* should be determined by dissolving the dehydrated product in water and titrating the solution at 50° C. with *N* sodium hydroxide solution, with phenolphthalein as indicator. The acidity, expressed as hydrochloric acid, should not exceed 1·5 per cent. Gelatin ash constantly contains iron, and the prescribed reaction with hydrogen sulphide is therefore always positive and does not allow of the recognition of other metals, particularly of copper. *Iron* should be estimated by moistening 1 grm. of ash with nitric acid, evaporating to dryness, heating to a dull red heat, adding 10 c.c. of 10 per cent. hydrochloric acid, and heating on a water bath until nearly dissolved. Evaporation is then carried to dryness, the residue treated with 20 c.c. of 5 per cent. hydrochloric acid, and the insoluble matter filtered off. Ammonium hydroxide is added to the filtrate until the precipitate is permanent, and, subsequently, acetic acid. The insoluble ferric phosphate is washed with distilled water until the filtrate gives no precipitate with ammonium oxalate, calcined and weighed, and the result multiplied by 52·98 to give the percentage of Fe_2O_3 in the ash. This should not exceed 2 per cent. *Copper.* The filtrate should not be coloured by the addition of 1 or 2 drops of 1 per cent. ferrocyanide solution. *Manganese* is nearly always present. Its amount should not be sufficient, on application of the Marshall-Bertrand process of dissolving 0·1 grm. of the ash in 10 c.c. of 0·25 nitric acid, adding 5 drops of 10 per cent. silver nitrate solution and 0·25 grm. of powdered potassium persulphate, and boiling, to produce a rose or violet coloration. The *ash* of the gelatin should not exceed 1·5 per cent. D. G. H.

Biochemical, Bacteriological, etc.

Preparation and Estimation of Methæmoglobin. M. Nicloux and G. Fontès. (*Comptes rend.*, 1924, **178**, 1757–1759.)—Treatment of blood with one-tenth of its volume of 95 per cent. alcohol constitutes a simple method of preparing methæmoglobin from the oxyhæmoglobin, the change occupying about six weeks at the ordinary temperature, or about a week at 37° C. The fact that alcohol prevents putrescence of blood and, hence, the conversion of methæmoglobin into hæmoglobin by the reducing processes accompanying putrescence renders it possible to manipulate methæmoglobin solutions in a medium closely comparable with blood and to estimate the pigment.

When blood (10 to 20 c.c.) containing a mixture of methæmoglobin and oxyhæmoglobin is shaken for an hour with pure carbon monoxide, the amount of the gas fixed is proportional to the amount of oxyhæmoglobin (a) present, since the other pigment does not combine with carbon monoxide. The gas dissolved physically is then removed in a vacuum, and the combined carbon monoxide extracted and estimated eudiometrically. The residual blood is treated with sodium hydrosulphite in dilute ammoniacal solution, both the oxyhæmoglobin and the methæmoglobin being thus converted entirely into hæmoglobin. The total hæmoglobin is then combined with carbon monoxide, as before, and its amount (b) estimated. The percentage of oxyhæmoglobin in the mixed pigments of the blood is given by $100\ a/b$, and that of methæmoglobin by $100\ (b-a)/b$.

T. H. P.

Chemistry of the Pyrimidines. O. Baudisch. (*J. Biol. Chem.*, 1924, **60**, 155–166.)—At one time it seemed as if the pyrimidines, uracil and cytosine, behaved entirely differently. On treatment, however, with ferrous bicarbonate and air and subsequent autoxidation of the colourless intermediate products, they formed the same brilliant lemon-yellow pigment possessing special chemical properties. This pigment-formation by such mild treatment is remarkable biologically, and also physically, since by the change the absorption of light is extended far into the visible spectrum. The yellow pigment is formed more quickly from cytosine than from uracil, due to the catalysing action of the ammonia produced in the cytosine experiment. In alkaline solution at 37° C., the pigment is weakly autoxidisable itself, since the yellow colour gradually disappears from the surface. It may also be obtained from a synthetic isobarbituric acid. It has not been analysed, but possibly there is a linking of two carbon atoms, 5 of the pyrimidine ring, forming a bimolecular pinacone-like compound. The pigment, characterised by a great stability toward both acids and alkalis, shows different chemical reactions according to whether one deals with weakly acid or weakly basic solution. In basic solution it reduces phosphomolybdic acid with the formation of an indigo blue colour and reduces ammoniacal silver nitrate solution slowly in the cold and quickly on heating. With diazobenzenesulphonic acid it gives an intense, but temporary, brilliant red colour. Methylene blue solutions are decolorised quickly, but an intense blue colour appears at the surface, due to the absorption of oxygen from the air. This reaction shows the reduction and oxidation power of the pigment in alkaline solutions. In acid solution the yellow pigment is very susceptible to reducing agents. Zinc dust easily decolorises it, but, on warming the colourless solution in an open bath, the yellow colour returns on autoxidation. The biochemical significance of the pigment is discussed, and details of the experimental work are given. It is assumed that the pyrimidines, uracil and cytosine, or their decomposition products, dihydroisobarbituric acid (not yet isolated), isobarbituric acid and the brilliant yellow compound possess the functions of respiration pigments. A deeper insight is obtained into the necessity of iron which is found in every cell nucleus. The system—ferrous bicarbonate plus air—causes the addition of one molecule of water to the ethylene group in uracil and in cytosine, giving dihydroisobarbituric acid. P. H. P.

Titration of Chlorides in Plant Fluids. J. V. Lawrence and J. A. Harris. (*J. Amer. Chem. Soc.*, 1924, **46**, 1471–1477.)—The results of tests of a wet oxidation and modified Volhard method for the rapid and accurate estimation of chlorides in plant tissue fluids are given. All the chlorides are precipitated by the addition of known excess of silver nitrate solution. Organic substances that introduce a source of error by fixing some of the silver added to the fluids, and pigments which preclude the detection of end-points in titration may be effectively and conveniently eliminated as sources of error by boiling with concentrated nitric acid. When silver chloride is thus precipitated from solution it is agglomerated into masses, as noted by Whitehorn (*J. Biol. Chem.*, 1921, **45**, 449), and titration of the excess of silver nitrate, with a standard solution of potassium thiocyanate, may be carried out in the presence of the precipitated silver chloride. Iron alum is used as an indicator. The solutions required and the analytical procedure are given and the accuracy of the method is discussed. Many hundreds of analyses have been made on a wide range of plant materials during the past three years. The method developed and used extensively for plant tissue fluids gives excellent results for blood chlorides, but is not recommended for this purpose because of the resistance of the hæmoglobin to the action of nitric acid, although, since this was written, Van Slyke (*J. Biol. Chem.*, 1923, **58**, 523) has suggested essentially the same method for blood. That it is applicable to fluids of much higher protein content than that found in plant saps is shown by the fact that it has already been used in the analysis of hides. P. H. P.

Toxicological and Forensic.

Solubility of Arsenic in Beverages. S. Goy and E. Wende. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 267–269.)—The amount of arsenic dissolved when solid arsenious oxide is added to liquid food depends upon the variety of arsenious acid used, as well as on the time and temperature, but is always much less than the recorded solubility figures for water. The following amounts of arsenious oxide per 100 c.c. were found in solution when the finely powdered amorphous substance was shaken with liquids for 3 minutes and filtered:

Coffee, 2 per cent. infusion,	temp. 72 C.	0·041 grm.
Coffee substitute, ,,	,,	0·04 ,,
Tea, 0·5 per cent. infusion	,,	0·04 ,,
Tap water	,,	0·26 ,,
Seltzer water, cold		0·013 ,,
Tap water ,,		0·016 ,,
Beer ,,		0·011 ,,

H. E. C.

Outbreak of Poisoning due to Cayenne Pepper containing Lead. M. Nicoloff. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 269–270.)—Adulteration of cayenne pepper is rife in Bulgaria, where much is used for seasoning food; the addition of maize-meal, sawdust and bran, with a dye, has been common, and, during a recent shortage, sunflower chips (that is, sunflower seeds after extraction of the oil) have been extensively used. In 1923 more than 1000 persons were affected by an outbreak of lead poisoning which was traced to adulteration of cayenne with 20 per cent. of red lead. H. E. C.

Potatoes containing a large amount of Solanine. E. Schowalter and W. Hartmann. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 251–257.)—Arising out of an outbreak of potato-poisoning in 1922 it is noted that the commonest physiological effect of solanine is to produce a constriction of the throat and sickness; it is also observed that partial immunity may be acquired. Experiments were made to find whether or not the use of potatoes with a high solanine content gives rise to a crop containing an excessive quantity when used for seed purposes, and these show that such seeds produce quite a normal content in the crop. There is no apparent connection between the type of soil or system of manuring and the solanine content; the high amount may have been due, in the year 1922, to the abnormal rainfall in the areas concerned. The increase in solanine content on storage is more than can be accounted for simply by loss of water, and appears to take place mainly in the outer layers of the potato, although it is not dependent upon the access of light. (*Cf.* also Bömer and Mattis, ANALYST, 1924, 284.) H. E. C.

Total Reduction of Nitrates and Chlorates during Putrefaction of Viscera. C. Ghigliotto. (*Ann. Chim. anal.*, 1924, **6**, 161.)—The viscera of two persons who had died after eating food which had been seasoned with sodium nitrate in error for sodium chloride were analysed six days after death when in a very advanced state of decomposition. They were tested for nitrate by Ogier's diphenylamine test, with a negative result. To establish whether or not nitrates could be entirely reduced during putrefaction, two flasks of nutrient broth were prepared similar to the suspected food and containing wheat-flour and beans, together with several drops of intestinal juice. To one flask were added 2 grms. of sodium nitrate per 700 c.c. of the broth, and to the other 10 grms. On testing both gave at once a positive result with diphenylamine. They were incubated at 37° C., and tested for nitrate at intervals. The flask containing 2 grms. gave a negative result at the end of 4 days ; that containing 10 grms., after 11 days.

A similar experiment was made with potassium chlorate, and it was found that whereas with nitrate a very fœtid odour was evolved, there was no disagreeable smell formed when chlorate was used (0·5 to 1 grm.), and a small white pellicle formed. When 0·5 grm. of potassium chlorate per 700 c.c. of broth was used the diphenylamine reaction was negative on the fourteenth day; with 2·5 grms. the result was negative on the twentieth day. R. F. I.

Water Analysis.

Estimation of Manganese in Water by the Sodium Bismuthate Method. W. D. Collins and M. D. Foster. (*Ind. Eng. Chem.*, 1924, **16**, 586.)—The following procedure is recommended for the estimation of manganese by the bismuthate method. A quantity of the water (100 c.c.) containing not more than 1 mgrm. of manganese is treated with 10 c.c. of dilute nitric acid (1:3) and 1 c.c. of concentrated sulphuric acid, evaporated, and the residual solution heated until most of the sulphuric acid has been expelled. The residue is dissolved in 50 c.c. of water and 20 c.c. of dilute nitric acid (through which air has been bubbled to remove lower nitrogen oxides), 0·1 grm. of sodium bismuthate is added, and the mixture is stirred for one minute and filtered through an alundum crucible or through an asbestos filter which has been washed with permanganate solution. The filtrate is diluted to a definite volume and its colour compared with standards. These are prepared by measuring out definite volumes of standard permanganate solution, adding to each the same quantity of nitric acid as that used for the sample, and diluting the mixtures to the same volume. W. P. S.

Organic Analysis.

Dehydration of Alcohol by means of Glycerin. E. Knecht and E. F. Muller. (*J. Soc. Chem. Ind.*, 1924, **43**, 177–178T.)—Distillation at a temperature not exceeding 80° C. of alcohol to which glycerin has been added in the proportion of 25 per cent. by weight of glycerin for 10 per cent. of water present, (20 per cent. for 5 per cent. of water, or 15 per cent. for 2 per cent. of water) followed by a second distillation with the use of smaller quantities of glycerin, was found to remove almost the whole of the water present. Rectification may be carried out in one operation, either by causing the distilled vapour, before reaching the condenser, to pass through 3 narrow wash bottles containing glycerin and heated to 78° C., or through an inverted upright condenser heated by a current of steam through the jacket and filled with glass beads over which glycerin trickles at the rate of 1 drop in 5 seconds. A 99·5 per cent. product is easily obtained at an additional cost of 4d. to 5d. per lb., and the process has the advantage of speed. The authors draw attention to the somewhat similar method recently proposed by Mariller for commercial dehydration of alcohol. (*Chim. et Industrie*, 1923, Oct.)

D. G. H.

Oxidation of Acetic Acid by different Metallic Chromates. L. J. Simon. (*Comptes rend.*, 1924, **178**, 1816–1819.)—Under the conditions employed in the author's method of estimating carbon in organic compounds (*cf.* ANALYST, 1922, **47**, 405; 1923, **48**, 37, 398), low results, and also an increased proportion of carbon monoxide, are obtained with acetic acid if the silver dichromate is replaced by an equivalent amount of another dichromate. T. H. P.

Estimation of Acetic and Butyric Acids. E. Fylemann (*J. Soc. Chem. Ind.*, 1924, **43**, 142–143 T.)—In fermented liquids butyric acid may be estimated with considerable accuracy by boiling the liquid with excess of potassium dichromate in a sulphuric acid solution of definite strength, the butyric acid being thus oxidised to acetic acid and carbon dioxide, and the dichromate being reduced to the extent of the equivalent of six atoms of oxygen. Propionic acid exerts a reducing action equivalent to three atoms of oxygen, and an allowance must be made for acetic acid, although this is almost unattacked.

The distillate containing the volatile acids is titrated as usual with 0·25 *N* sodium hydroxide solution (*n* c.c. required) in presence of a minimum quantity (2 drops) of ordinary alcoholic phenolphthalein, and is then boiled to one-half of its volume in order to eliminate the alcohol and afterwards diluted until 20 c.c. of the liquid correspond with about 1 c.c. of 0·25 *N* sodium hydroxide. Twenty-five c.c. of 0·25 *N* potassium dichromate are added to 20 c.c. of the liquid in a 300 c.c. conical flask, to which is fitted a reflux condenser; 30 c.c. of 96 per cent. sulphuric acid are then introduced by way of the condenser, the flask being shaken meanwhile. The liquid is at once boiled for exactly an hour, and then cooled, diluted to about 250 c.c., mixed with potassium iodide, and titrated with 0·25 *N* thiosulphate solution.

Apart from the correction for acetic acid, 1 c.c. of 0·25 *N* dichromate solution, which is equivalent to 8 grms. of available oxygen per litre, represents $80 \div (6 \times 8000) = 0{\cdot}00183$ grm. of butyric acid. Should propionic acid be present, this cannot be distinguished from butyric acid by this method, but as the oxygen absorbed is proportional to the increased weight of organic acids, the total weight of organic acids is correctly determined. Since the oxidation to acetic acid occurs mostly in a very few minutes, the correction for this acid may be based on the organic acid originally present, that is, on the total acidity, and is equal to 0·5 *n*. Thus, if *b* c.c. of dichromate are reduced, 0·00183 ($b-0{\cdot}5\,n$) grms. of butyric acid are present in the amount of distillate employed. In the case of propionic acid, the amount is 74 ($b-0{\cdot}5\,n$) $\div (3\times 8000) = 0{\cdot}00308\,(b-0{\cdot}5\,n)$. T. H. P.

Estimation of Calcium Carbide. J. Y. Yee and H. J. Krase. (*J. Amer. Chem. Soc.*, 1924, **46**, 1389–1393.)—An accurate method has been developed for the estimation of carbides that yield acetylene when treated with water, by the application of the method of Chavastelon, as modified by Ross and Trumbull (*J. Amer. Chem. Soc.*, **1919**, **41**, 1180) for the analysis of gas mixtures containing acetylene and ethylene. Chavastelon's method involves the absorption of acetylene in neutral or ammoniacal silver nitrate. With the neutral solution and an excess of silver nitrate the reaction is quantitative according to the equation

$$C_2H_2 + 3AgNO_3 = C_2Ag_2.AgNO_3 + 2HNO_3 \qquad (1).$$

It is also quantitative with ammoniacal silver nitrate,

$$C_2H_2 + 2AgNO_3 + 2NH_4OH = Ag_2C_2 + 2NH_4NO_3 + 2H_2O \qquad (2).$$

In the first reaction the liberated nitric acid can be directly titrated, and in the second the excess of silver nitrate must be found. Both methods are satisfactory for carbides, the former being the more rapid of the two, but for carbides containing nitrides reaction (2) must be employed. Full experimental details are given; also precautions necessary and a diagram of the apparatus required. Traces of sulphides do not interfere when proper precautions are taken. Phosphides, however, should be absent. Some results given shew that the two methods agree satisfactorily. P. H. P.

The Sodium Plumbite Test of Gasoline. B. T. Brooks. (*Ind. Eng. Chem.*, 1924, **16**, 588.)—In this test the gasoline (light petroleum) is shaken with litharge dissolved in sodium hydroxide solution; if the sample contains sulphur compounds a yellow coloration develops. A very small quantity of sulphur is then added and the mixture again shaken, when the colour is much intensified, a flocculent precipitate forms, and the colour changes rapidly from dull orange to brown and finally brownish-black. Although practically all the gasoline refined to-day is free from sulphur compounds and does not give a coloration with the test, certain specimens which have been kept for a few months yield a coloration and a brownish precipitate; this is due to the formation of organic peroxides in the gasoline, and the precipitate consists of lead peroxide. In these cases, the addition of sulphur does not intensify the colour. If allowed to stand for a few hours, the precipitate due to sulphur compounds becomes quite black, whilst the lead peroxide precipitate retains its brownish shade. W. P. S.

New Form of Fenchone Oxime. Characterisation of Fenchone in presence of Camphor. M. Delépine. (*Comptes rend.*, 1924, **178**, 1721–1723.)—Fenchone in presence of camphor may be readily detected by converting the mixed compounds into their oximes and taking advantage of the greater solubility and more marked acidic character of camphor oxime. A solution of a mixture of equal amounts of the two oximes in 5 to 7 times their weight of boiling alcohol deposits one-half of the fenchone oxime in crystals having $[\alpha]_D = +45°$, fresh crystals with $[\alpha]_D = +47°$ being obtained on addition of potassium hydroxide and water to the mother liquor; acidification of the alkaline liquid by means of acetic acid then yields camphor oxime with $[\alpha]_D = -40°$. Two new fenchone oximes (β) have been prepared, one dextro-rotatory and one lævo-rotatory. There are thus six fenchone oximes known, the α- and β-oximes existing in dextro-, lævo-, and racemic modifications. T. H. P.

New Reaction for Low Temperature Tars. F. Schulz and J. Prunet. (*Bull. Soc. Chim.*, 1924, **35**, 638–641.)—When 1 to 2 c.c. of a lignite tar, or any of the oils obtained therefrom by distillation, are shaken for about a minute with 5 c.c. of a solution containing 25 grms. of crystallised potassium oxalate per 100 grms. of solution and treated just before use with a few drops of ferric chloride solution, the lower layer which separates on standing assumes an indigo coloration. This reaction, which is due largely to the catechol present in the tar or tar oil, is not shown by aromatic coke-oven or gasworks tars or their distillates, but is given by tar obtained by distilling certain coals at low temperatures in glass retorts, the substances determining the reaction apparently undergoing destruction at a high temperature. T. H. P.

Estimation of Glycerol by Oxidation with Iodate and Sulphuric Acid. R. Strebinger and J. Streit. (*Zeitsch. anal. Chem.*, 1924, **64**, 136–143.)—Potassium iodate and strong sulphuric acid oxidise glycerol quantitatively, and the excess of iodate can be estimated iodimetrically. The percentage of glycerol is given by the expression $\frac{92{\cdot}1\,(a-b)\,100}{599{\cdot}26\,E}$, in which a = iodate added b = remaining iodate, and E = weight of substance taken. About 0·2 to 0·3 (or, with dilute solutions a quantity containing 0·025 to 0·05) grm. of glycerol, 8 to 9 times its weight of potassium iodate (accurately weighed), and 30 c.c. of pure, strong sulphuric acid are heated in a conical 300 c.c. flask on a sand-bath; in the case of dilute solutions the quantity of sulphuric acid must be increased to 50 c.c. Copious evolution of iodine vapour sets in at 170° C. The temperature is gradually raised until the iodine is expelled and white fumes appear. The acid retains a little iodine, which colours it green. When cold, it is diluted with water, when the colour changes to brown. The liquid is boiled till colourless, again diluted and made up to volume in a graduated 250 c.c. flask. Fifty c.c. are titrated, after addition of 1 to 2 grms. of potassium iodide, with 0·1 N thiosulphate solution standardised against potassium iodate. For the estimation of glycerol in fats, 2 to 3 grms. are saponified with 5 grms. of caustic alkali in 30 c.c. of water and 10 c.c. of methyl alcohol for 2 to 3 hours under reflux. The alcohol is expelled on the water-bath, and the hot liquid treated with dilute sulphuric acid, and cooled. The fatty acids are filtered off on a wet double paper, and well washed, and the filtrate evaporated to one-half its bulk on the water-bath. If this causes a precipitate of albuminous matter (as in the case of tallow), the liquid is again filtered through a wet paper, which is subsequently well washed. The filtrate is ready for the estimation of the glycerol. W. R. S.

Tung Oil. Oil from the Seeds of *Aleurites montana*. R. N. Parker, M. G. Rau, W. A. Robertson, and J. L. Simonsen. (*Indian Forest Rec.*, 1924, **10,** 1–13.)—Some confusion exists in the literature of the subject as to the species of *Aleurites* from which Chinese wood oil is obtained; Hankow wood oil is derived from the seeds of *A. Fordii* and that from Canton from *A. montana*; the botanical characters of these seeds and leaves are described and illustrated. The oil from Southern Japan, which is derived from *A. cordata*, is not exported to Europe. Examination of the oil from *A. montana* shows it to consist of the glycerides of β-elaeostearic, oleic, and probably linolic acids, whereas Fahrion has shown that the oil from *A. Fordii* consists of glycerides of α-elaeostearic and oleic acids. The following table gives the data obtained in the analysis of oil from these species, and a comparison with the proposed American standard.

	A. montana.	*A. Fordii.*	American specification.
Specific gravity at 15°/15° C.	0·9467	0·9439	0·939–0·943
Refractive index, n_D^{15}	1·494	1·4917	n_D^{25} 1·515–1·520
Acid value	3·4	nil	7·0 max.
Saponification value	203	204	190–195
Unsaponifiable matter, per cent.	0·6	—	0·75
Iodine value	141·4	157·7	163
Heat test	16′	12·5′	12′
Iodine jelly test	45″	35″	—
"Titre test"	54–54·5°	—	—

The heat test is considered the most satisfactory criterion. H. E. C.

Phytosterols of the Endosperm of Maize. R. J. Anderson. (*J. Amer. Chem. Soc.*, 1924, **46,** 1450–1460.)—The unsaponifiable matter derived from the endosperm of maize contains at least two fractions of phytosterol that differ in properties and composition. The endosperm of maize contains some free phytosterol; m.p., 137–137·5° C.; specific rotation $-32{\cdot}23°$; acetyl derivative, m.p., 127° C. After saponification, the unsaponifiable matter was separated into the following 3 parts: (1) The optically active dihydrositosterol, $C_{27}H_{47}OH.H_2O$; m.p., 138–139° C. The dried preparation melts between 140 and 141° C.; $[\alpha]_D^{20}, +25°$; acetyl derivative, m.p. about 138° C.; $[\alpha]_D^{20}, +14{\cdot}41°$. (2) Rather large quantities of the ordinary sitosterol associated with the dihydrositosterol in the endosperm and bran of maize. (3) A brownish-yellow oily substance that has not been examined. Dihydrositosterol crystallises in the same form as sitosterol, but the crystals are larger and denser. It does not give the Liebermann-Burchard reaction, and the Whitby reactions are not typical. It does not absorb bromine. All the experimental work is described in detail, and results of combustions are given. The laevorotatory sitosterol and the dextrorotatory dihydrositosterol exist in various plant fats. The author finds that the magnitude of the optical rotation is a safer and more reliable criterion of purity in these compounds than is the melting point. Plants may elaborate several different phytosterols, and these may be deposited in special parts of the plants. P. H. P.

Substitutes for Mercuric Chloride in Hubl's Iodine Solution. B. M. Margosches and W. Hinner. (*Chem. Zeit.*, 1924, **48**, 389–391.)—The influence of mercuric chloride and the various substitutes which have been proposed for it in connection with the estimation of the iodine value of oils has been experimentally studied. The activation of an alcoholic iodine solution by mercuric chloride depends on the formation of mercuric iodide and iodine monochloride, and a similar effect is produced by cadmium chloride; the mercury, as such, has no specific action. In the case of Ley's solution of iodine and mercuric acetate in acetic acid there is similarly the production of mercuric iodide and acetic acid from the hydriodic acid formed from the fat. In the solution of iodine and mercuric iodide in methyl alcohol, proposed by Gill and Adams, there is formed an even greater quantity of acid than with Hubl's solution; the accelerating effect of the mercuric iodide is mainly due to its combination with the hydrogen iodide formed during the reaction, which would otherwise retard it. H. E. C.

Bromometric Estimation of Iodine Values. W. Manchot and F. Oberhauser. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 261–263.)—The authors confirm the reliability of Rosenmund and Kuhnhenn's method, in which the reagent is pyridine sulphate bromide, $C_6H_5N.H_2SO_4.Br_2$ (*cf.* ANALYST, 1924, 105), and show that a 0·1 *N* solution of bromine in 20 per cent. hydrochloric acid also gives accurate results when using chloroform as solvent and allowing a time of 4 hours. Similar results are obtainable in 12 hours in the absence of chloroform. As an alternative, bromine in technical (99 per cent.) acetic acid is satisfactory. A mixture containing 0·2 per cent. of indigo carmine and 0·2 per cent. of trinitroresorcinol is a more satisfactory indicator than is methyl orange in the final titration, which is carried out as described by Rosenmund and Kuhnhenn. H. E. C.

Nitrosomethylurethane as a Reagent for Catechol Tans. W. Vogel and C. Schüller. (*Coll.*, 1923, **642**, 319.)—Ten to twelve c.c. of clear filtered tannin solution of analytical strength are treated in a test-tube with 3 to 5 drops of 10 per cent. hydrochloric acid. The solution should remain clear on boiling. To the boiling solution are added 5 to 7 drops of nitrosomethylurethane, and the boiling continued for 3 to 4 minutes. The solution must be allowed to cool slowly, when, if catechol tans are present, a precipitate is formed. The precipitation is, in general, characteristic for catechol tans. Pyrogallol tans give no precipitate. The above reaction is five times as sensitive as the bromine-water test, and possesses the further advantage that it is not influenced by the presence of sulphited tans, synthetic tans or cellulose extracts. R. F. I.

Microchemical Tests for Hexamethylenetetramine. H. Leffmann. (*Amer. J. Pharm.*, 1924, **96**, 366–367.)—A small fragment of the sample is dissolved in a drop of water; a drop of concentrated hydrochloric acid and 2 drops of a magenta and sulphurous acid solution are then added. If hexamethylenetetramine is present a bluish tint develops, gradually deepening in intensity. This test is suggested as more convenient than, and as delicate as Denigès' method (*Bull. Pharm. Bordeaux*, 1924, **62**, 3) which consists in the microscopical examination of the residue from the solution of the fragment of sample in 1 volume of alcohol and 2 volumes of chloroform, and subsequent recrystallisation with various reagents. D. G. H.

Inorganic Analysis.

Separation of Mercury from Cadmium by means of Pyridine. G. Rotter. (*Zeitsch. anal. Chem.*, 1924, **64**, 102–106.)—The neutral solution is treated with excess of ammonium thiocyanate, warmed gently, and treated, after cooling, with one-third of its volume of pyridine. After standing for at least one hour the heavy white crystalline precipitate of cadmium pyridine thiocyanate —$[Cd(C_5H_5N)_2](CNS)_2$—is filtered off and washed on a paper previously moistened with a solution of thiocyanate and pyridine. Re-precipitation is recommended. The precipitate is dissolved in a minimum of hydrochloric acid (1:10); the solution is neutralised with ammonia, and again treated with thiocyanate and pyridine. After standing as before the precipitate is filtered off, washed with the above washing-solution, dissolved in nitric acid, the solution evaporated to dryness in a tared crucible, and the residue ignited. After treating with sulphuric acid, the cadmium sulphate is weighed. Mercury is precipitated in the combined filtrates as sulphide. W. R. S.

Separation of Cadmium from Zinc. K. Chalupny and K. Breisch. (*Chem. Zeit.*, 1924, **48**, 349–351.)—The solution is neutralised with potassium hydroxide until a slight permanent precipitate is obtained; this is dissolved by addition of potassium cyanide solution (100 grms. in 300 c.c. water), followed by an excess of the same solution equivalent to 4 grms. of salt per grm. of zinc. The clear liquid is made alkaline with 10 c.c. of 4 *N* potassium hydroxide, and heated; at the boiling point 0·5 to 1 grm. of solid thio-urea is added, and boiling is continued for 30 minutes after the appearance of the precipitate of cadmium sulphide. This is filtered off at once and washed several times with hot dilute cyanide, and then with hot ammonium nitrate solution. The washed precipitate is dissolved in hot dilute hydrochloric acid, and the precipitation repeated in exactly the same manner. The authors estimate the cadmium by electrolysis; the sulphide is dissolved as before and the hydrogen sulphide boiled off; the solution is treated with potassium hydroxide until the appearance of a slight permanent precipitate. This is dissolved in a very small excess of cyanide, and the solution is electrolysed at 80° C. (5V, 5 Amp.), 0·05 grm. of metal being deposited in 30 to 45 minutes. The results are accurate. For the estimation of cadmium in spelter, the solution of a suitable quantity of metal is digested with 0·5 grm. of purest zinc filings at 80° C. for 2 hours. The precipitate is filtered off, dissolved in hydrochloric acid, and the cadmium estimated as described. W. R. S.

Use of the Lead Cathode in the Electrolytic Method for the Estimation of Minute Quantities of Arsenic. T. Callan. (*J. Soc. Chem. Ind.*, 1924, **43**, 168–170T.)—A series of experiments was carried out with a view to elucidating the erratic behaviour of lead cathodes continuously used in the electrolytic estimation of arsenic. The two main ways in which such cathodes may lose their sensitiveness were found to be: (1) By prolonged use in dilute sulphuric acid solutions, possibly due to a coating of a film of lead sulphate or oxysulphate. Inactivity in such cases is removed by treatment with dilute nitric acid, and it is strongly recommended that before every test lead cathodes be dipped for a few minutes in dilute nitric acid (with immediate removal on evolution of gas) and well washed. (2) By removal of traces of metallic impurity from the surface; this may be remedied by the addition of a trace of cadmium to the cell immediately before the addition of the solution to be tested. (*Cf.* ANALYST, 1922, **47**, 535.)

D. G. H.

Detection and Estimation of Antimony by means of Pyrogallol. F. Feigl. (*Zeitsch. anal. Chem.*, 1924, **64**, 41–47.)—Pyrogallol yields with antimonious salts a white, silky, crystalline precipitate of the composition $SbC_6H_5O_4$ (Sb 46·01 per cent.). The reaction takes place quickly and quantitatively in moderately acid solution; tartrate solutions are most suitable. The sensitiveness of the reaction is 1 in 217,400. For quantitative estimations the solution of antimony trichloride is treated with an equivalent amount of Rochelle salt and about five times the necessary amount of pyrogallol dissolved in 100 c.c. of water. The volume is made up to 250 c.c., the precipitate collected, after 2 hours' standing, on a Gooch crucible, washed with cold water, dried, and weighed. The process allows of a separation from arsenic, which is not precipitated, and may be recovered from the filtrate as sulphide. W. R. S.

Volumetric Estimation of Tin. J. G. F. Druce. (*Chem. News*, 1924, **128**, 273.)—The method of Knop for the titration of ferrous iron with dichromate, with diphenylamine as indicator (*J. Amer. Chem. Soc.*, 1924, **46**, 263), was extended to the estimation of stannous salt. The solution was treated with excess of ferric salt, and the ferrous salt formed titrated with 0·1 *N* dichromate solution; a drop of a solution of one grm. of diphenylamine in 100 c.c. of strong sulphuric acid served as indicator. Fifteen c.c. of acid mixture (equal volumes of strong sulphuric and phosphoric acids) were added where large amounts of dichromate were required, the end-point being marked by the appearance of a blue coloration. It was found feasible to titrate stannous salt in the manner described, without previous addition of ferric salt. According to the results given, the new method gives substantially higher results than the iodine method. W. R. S.

Precipitation of Platinum and Copper as Sulphides. R. Doht. (*Zeitsch. anal. Chem.*, 1924, **64**, 37–40.)—The gently boiling platinum solution, free from nitric acid, and containing 5 c.c. of hydrochloric acid, is treated with a 10 per cent. solution of sodium hypophosphite, then, while being constantly stirred, with an

aqueous solution of sulphur dioxide. The platinum is precipitated as sulphide, which is allowed to settle on the water-bath, filtered, washed with hydrogen sulphide water containing ammonium nitrate, and ignited strongly to metal. Copper solutions, when treated in the same manner, also yield a precipitate of sulphide. It is assumed that the sulphur dioxide is first converted into hyposulphurous acid, which is reduced further as follows: $H_2S_2O_4 + 2H_3PO_2 = 2H_3PO_4 + H_2S + S$.
W. R. S.

Separation of Zinc by Electrolytic Deposition from Alkaline Solutions K. Breisch. (*Zeitsch. anal. Chem.*, 1924, **64**, 13–23.)—Nitrates interfere with the electrolysis of zinc in alkaline solution, as deposition does not begin until they have been completely reduced to ammonia. It was found that the nitrates can be eliminated as follows: The solution (150 c.c.) is heated to boiling with 50 c.c. of strong hydrochloric acid, and treated with small quantities of solid paraformaldehyde until red fumes are no longer evolved; the oxidation is complete in a few minutes. The solution is cooled, made alkaline, and heated with hydrogen peroxide (perhydrol) to destroy the paraformaldehyde; boiling decomposes the excess of hydrogen peroxide. In the test experiments described silver gauze cathodes were used; they were cleaned by digestion in cold strong hydrochloric acid. A number of rapid separations can be carried out in alkaline solution by electrolysing with a current of 3 Amp. and 4 to 5 V. with a rotating stirrer (500 R.P.M.); 0·25 grm. of zinc is deposited in 30 minutes. *Separation of Zinc from Nickel.*—The solution (less than 100 c.c.), to which 5 c.c. of perhydrol has been added, is slowly poured into a hot 50 per cent. solution of 20 to 25 grms. of potassium hydroxide, heated a short time till effervescence ceases, and electrolysed. The nickel hydroxide is subsequently dissolved in acid, and the solution electrolysed or titrated. *Zinc from Aluminium.*—The separation from large quantities of aluminium is quite satisfactory; the electrolysis is carried out as for a pure zinc solution, sufficient alkali being added to keep the alumina in solution. *Zinc from Iron (Aluminium).*—The solution is treated with sodium sulphite to reduce iron, made alkaline with potassium hydroxide, and the precipitate dissolved by cautious addition of 20 per cent. cyanide solution from a burette. Soon after the beginning of the electrolysis a saturated solution of 10 grms. of sodium sulphite is added; a second addition is required after 25 to 30 minutes (for $ND100 = 3A$); anodic formation of ferricyanide is thus prevented. W. R. S.

Estimation of Manganese by means of Persulphate and Silver Nitrate. K. Swoboda. (*Zeitsch. anal. Chem.*, 1924, **64**, 156–184.)—A detailed study was made of the volumetric estimation of manganese in steel by oxidation to permanganic acid with persulphate and silver nitrate, followed by titration with arsenite. As a result, the following modification is recommended: 0·25 grm. of steel (0·5 grm. if manganese is below 0·5 per cent.) is dissolved in a 500 c.c. conical flask, in 25 c.c. of acid mixture (water 1000, nitric acid 250, sulphuric acid 40 c.c.). The red fumes are boiled off; 10 c.c. of 0·1 *N* silver nitrate solution, enough water to bring the volume to 200 c.c., and 4 c.c. of ammonium persulphate solution

(500 grms. per litre) are added. The mixture is boiled for 3 to 5 minutes to decompose the persulphate, the liquid cooled, and 20 c.c. of 10 per cent. potassium fluoride solution or 1·5 c.c. of strong hydrofluoric acid added. The solution is immediately titrated with the arsenite solution (0·5 grm. of arsenic trioxide and 1·5 grm. of sodium bicarbonate per litre) until colourless, or, with high manganese contents, faintly green. A steel of known manganese content is taken as the standard. The end-point in this procedure is much more easily observed; the volume of arsenite solution reacting with the permanganic acid is only four-fifths of that consumed in the usual process, the reduction proceeding to the stable manganic fluoride. W. R. S.

Estimation of Manganese. Manganous Oxalate as a Standard. R. W. Coltman. (*Ind. Eng. Chem.*, 1924, **16**, 606–609.)—Manganous oxalate is proposed as a standard in the estimation of manganese by Volhard's method, the chlorate method, and similar processes. To prepare the salt, 250 grms. of pure crystallised oxalic acid are dissolved in about 500 c.c. of hot water and a solution of 60 grms. of pure potassium permanganate is added slowly while the oxalic acid solution is kept near the boiling point. The permanganate colour disappears quickly after each addition and, finally, manganous oxalate begins to precipitate. When all the permanganate has been added the hot supernatant liquid is decanted, the precipitate washed with cold water until free from oxalic acid and then dried in a current of dry air. The salt has the composition $MnC_2O_4.2H_2O$; it is not hygroscopic, does not effloresce, and will keep indefinitely in stoppered bottles under ordinary conditions. If the oxalic acid solution is allowed to become too cold during the addition of the permanganate, some trihydrate ($MnC_2O_4.3H_2O$) may form; in this case the washed salt must be heated with water at 90° C. until all pink colour is destroyed, and the salt again collected, washed and dried. If manganous oxalate is heated with sulphuric acid until fumes of the acid are evolved, manganous sulphate for use in Volhard's method is obtained, whilst treatment with concentrated nitric acid will give a solution of manganous nitrate for the chlorate or bismuthate method. W. P. S.

Diphenylamine Indicator with Iron. W. W. Scott. (*J. Amer. Chem. Soc.*, 1924, **46**, 1396–1398.)—Diphenylamine indicator, shown by Knop (*J. Amer. Chem. Soc.*, 1924, **46**, 263) to be valuable in the titration of iron with potassium dichromate, may also be used in potassium permanganate titrations of iron. Hydrochloric acid, stannic chloride and mercurous chloride, if present, do not interfere, thus enabling potassium permanganate titrations to be made where formerly they were not possible. As suggested by Knop, several c.c. of a phosphoric and sulphuric acid mixture (15 c.c. of each increased to 100 c.c. with water) were used by the author to repress the yellow colour of (ferric) chloride. The delicacy of the reaction in permanganate titrations depends on a judicious use of the indicator. The quantity of indicator used should not exceed 0·4 c.c., and the best results are obtained by the use of 0·2 c.c. of the indicator, made according to Knop's directions (1 grm. of salt per 100 c.c. of concentrated sulphuric acid) in solutions of iron in volumes of 100 to 250 c.c. If solutions are diluted too much, the end-point is a violet in place of a deep blue colour. A table of results is given. Diphenylamine indicator may be used in the standardisation of potassium dichromate by means of standard ferrous sulphate solution, since titrations may be done with either reagent without destroying the qualities of the indicator, the blue colour disappearing with an excess of ferrous salt, but being restored by an excess of dichromate. P. H. P.

Mechanical Separation of Polonium in Sodium Hydroxide Solution by Various Substances. J. Escher-Desrivières. (*Comptes rend.*, 1924, **178,** 1713–1715.)—The mechanical separation of polonium, present in sodium hydroxide solution, by an insoluble hydroxide, such as that of bismuth, and its adsorption by a particular sample of animal charcoal, depend on the number of molecules of sodium hydroxide contained in a given volume of the mixture. Traces of the hydroxide are sufficient to remove all the polonium from its solution in decinormal sodium hydroxide; the active element also undergoes rapid deposition on the walls of the containing vessel. When, however, the concentration of the sodium hydroxide is high, the polonium remains in solution. T. H. P.

Colorimetric Estimation of Nitrate Nitrogen. F. M. Scales and A. P. Harrison. (*Ind. Eng. Chem.*, 1924, **16,** 571–572.)—The method depends on the formation of a red coloration when a nitrate is treated with strychnine in the presence of sulphuric acid and mercuric chloride. To prepare the strychnine reagent, equal volumes of 0·5 per cent. strychnine sulphate solution in concentrated hydrochloric acid and 0·1 per cent. mercuric chloride solution in water are mixed; 25 c.c. of this mixture are added slowly to 1 grm. of magnesium powder and, when reaction has ceased, the solution is cooled and filtered. The reagent should be used within a few hours of its preparation. One c.c. of the reagent is placed in a test-tube, 5 c.c. of the test solution (*e.g.* soil extract) are added, followed by 5 c.c. of concentrated sulphuric acid; the mixture is then poured into another test tube, and the coloration obtained is compared with those produced by known amounts of nitrate treated similarly and at the same time. The comparisons need not be made immediately, but all the mixtures must be made within five minutes of each other, as the colour deepens slowly on standing. Comparative results obtained by this method and by titration as ammonia are given, the amounts of nitrate nitrogen ranging from 0·2 to 4·6 mgrms. W. P. S.

Application of the Aldehyde-Bisulphite Reaction in Volumetric Analysis A. Kurtenacker. (*Zeitsch. anal. Chem.*, 1924, **64,** 56–61.)—The formation of the aldehyde-bisulphite compound was found to be applicable to volumetric work in cases where the iodimetric estimation of another compound is required in presence of a sulphite. Thiosulphate may be thus titrated; two procedures were worked out. (1) If the solution is neutral towards methyl orange (*i.e.* contains bisulphite), it is treated with 5 c.c. of 35 per cent. formaldehyde, and titrated, after 5 minutes, with iodine solution. The end-point with starch is very sharp and persists several minutes. Acid or alkaline solutions are first neutralised towards methyl orange; as this indicator interferes, the required quantity of acid or alkali is ascertained in another portion. (2) The solution, if acid, is neutralised towards phenolphthalein (conversion into sulphite). Formaldehyde is added, and the deep red colour then discharged with 10 per cent. acetic acid, of which an excess of 20 c.c. is added; the solution is immediately titrated with iodine. The author prefers the second method. The results given are very good.

W. R. S.

Iodimetric Estimation of Phosphorous and Hypophosphorous Acids. A. Brukl and M. Behr. (*Zeitsch. anal. Chem.*, 1924, **64**, 23–28.)—The solution of one of the acids, free from hydrochloric or nitric acid, is acidified with sulphuric acid and boiled with *a* c.c. of 2·5 per cent. solution of iodic acid (standardised against thiosulphate) until it is colourless and the liberated iodine completely expelled. After cooling, the excess of iodic acid is ascertained by addition of potassium iodide and titration with thiosulphate (*b* c.c.):

$$15H_3PO_2 + 12HIO_3 = 15H_3PO_4 + 6H_2O + 12I; \text{ and}$$
$$15H_3PO_3 + 6HIO_3 = 15H_3PO_4 + 3H_2O + 6I.$$

The quantity of iodic acid consumed by the phosphorous (hypophosphorous) acid is equivalent to one-sixth of that of the iodine indicated by $(a - b)$. For the separation and estimation of the acids in mixtures, the solution is evaporated to dryness with a slight excess of alcoholic potassium hydroxide; the residue is transferred to a graduated 50 c.c. flask by means of alcohol. After standing for two hours, with frequent stirring, the solution is made up to the mark, filtered through dry paper, and the hypophosphorous acid estimated iodimetrically in an aliquot part of the filtrate after evaporation of the alcohol. Phosphorous acid is obtained by difference. (Potassium phosphite is soluble in alcohol to the extent of 0·0007 grm. of H_3PO_3 per 10 c.c.).

W. R. S.

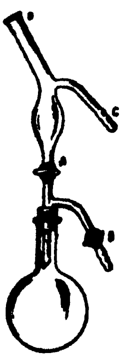
Physical Methods, Apparatus, etc.

Melting Point of Coal Ash. F. S. Sinnatt and N. Simpkin. (*Lanc. and Cheshire Coal Res. Assoc. Bull.*, 15, 1924.)—A method has been devised for the determination of the m.p. of coal ash, so that it may be carried out on as little as 0·2 grm. of the ash. The coal is ground to pass a No. 60 sieve and incinerated in a muffle at 800–850° C., care being taken to exclude access of the products of combustion of coal gas; then the ash is ground to pass a No. 200 sieve and moistened in an agate mortar with a 10 per cent. solution of dextrin. The paste so formed is pressed through a screw press having an orifice $\frac{1}{32}''$ diameter, so that about 1″ protrudes; then it is dried on a hot plate at about 250° C. and cut into pieces $\frac{3}{16}''$ in length. The furnace consists of a length of 6 inches of vitreosil tube, set in a steel-covered magnesia brick having a central aperture, so that the tube can be heated directly by an oxygen and coal-gas blow pipe. The temperature, which is regulated by controlling the oxygen supply, is observed by means of a thermocouple placed about $\frac{1}{16}''$ from the rod of ash; the m.p. is observed directly.

The m.p. of the ash from the separated four constituents of coal—vitrain, clarain, fusain, and durain—differs in different seams and in different horizons in the same seam within wide limits, and bears no apparent relation to its chemical composition. H. E. C.

Identification of Phenols by means of the Spectroscope. S. Palkin and H. Wales. (*J. Amer. Chem. Soc.*, 1924, **46**, 1488–1493.)—The diagnostic value of absorption spectrum examination as a supplement to colour tests is pointed out. Azo dyes can be prepared easily with the use of the reagent diazo-*p*-nitrobenzene, which, when properly prepared, has been kept in a refrigerator for months without decomposition. The absorption spectra of azo dyes prepared from certain phenols, by coupling with azo-*p*-nitrobenzene, have been examined in three solvents, *viz.* alkaline (1) acetone, (2) alcohol and (3) water, and the data comprising absorption curves and tables of absorption-spectrum maxima are recorded for reference. The authors discuss the application of this procedure to the detection and the identification of small amounts of phenols, and a method adapted particularly to the examination of medicinal preparations is outlined. Less than 0·01 mgrm. of the phenols examined has been detected. P. H. P.

Apparatus for Fractional Distillation under reduced Pressure. E. André. (*Bull. Soc. Chim.*, 1924, **35**, 647–648.)—This apparatus consists of a small ellipsoidal vessel the neck of which, D, is inclined and is connected with the condenser. At the side is a tubulure, C, connected with a water-pump, and at the base a tap, A, communicating with a tube on which is a side tubulure, B, also connected with a water-pump. In making a distillation the tap A is opened and the tap B closed when a large enough fraction has been collected in the small receiving flask. The tap A is then closed, and the tap B (temporarily disconnected from its water-pump) is opened, so that the air enters the receiving flask, which can then be detached. After its contents have been transferred to another vessel, the receiving flask is replaced, and the air again exhausted from it, by means of the second water pump, through the tap B. This tap is then closed, the tap A opened, and the distillation resumed. The small amount of liquid distilling during the change will have collected in the vessel above A.



Reviews.

Les Isotopes. Par A. Damiens. Pp. ix.+114. Paris: Gauthier-Villars et Cie. 1923. Price 12.50 fr.

Most of those who have lived since the time of Anaxagoras have concerned themselves but little with the constitution of matter Epicurus, Lucretius, possibly the author of Genesis ii. 7, and Newton are honourable exceptions to this widespread lack of interest, but it was reserved for Dalton to elaborate and systematise the conception of atoms adumbrated by these authors. That matter was continuous rather than discrete has been argued by many, and Ostwald as recently as 1903 maintained in his Faraday lecture a non-objective theory of matter. Now the atom and the quantum—the atom of action—are regarded as real entities.

The notions of atoms and elements are closely linked, and the widespread acceptance of Boyle's definition that "that was to be regarded as elementary which could not by any means be separated into different substances," and of Lavoisier's statement that one might define an element as "une substance dont le poids ne peut qu' augmenter, au cours des transformations chimique qu'il subit" led to a theoretical conception of the element, which has been found, after over a century of atomic theory, to be, in some cases, untenable.

In the 19th century chemists were so much occupied in studying the interrelations of atoms that but little curiosity was expressed as to the possibility of a complex structure of the minute infinitely hard sphere itself. The hypothesis of Prout which Stas regretfully believed that he had disproved, and the Periodic Law, the soundness of which was obvious, notwithstanding many discordant facts, were the outcome of a strong subconscious feeling in favour of the unity of matter and the necessary complexity of atoms. The discovery of spontaneously disintegrating elements and other disturbing but undoubted facts diverted the attention of mathematically minded chemists and physicists from the study of matter as such to an investigation of the atom itself, and led Rutherford and Bohr to a conception of its structure, now, in the main, accepted. This, while allowing the infinitely hard sphere to remain, showed that it must be so considered by reason, not of a solid continuity, but of the very great speed of rotation of its outer planetary electrons around a central nucleus. This speed, although too great to allow atoms to deform one another, nevertheless allows the vast majority of smaller faster-moving particles which may bombard an atom to pass through the relatively vast space within it, although some, and these the most interesting, are diverted from their linear motion by collision, attraction or repulsion, with or by the nucleus.

The work before is, as M. Perrin in his preface says "clair et agréable à lire." It sets forth the facts concerning those elements which varied evidence leads us to consider as mixtures of substances having identical chemical properties, and which Soddy has happily called *isotopes* (ἴσος same, τόπος place), as the constituents occupy the same place in the periodic table. In his first and second chapters the author relates these facts concerning the groups of radio-active elements derived respectively from uranium and thorium, showing that the end-product of each series should be, and is, lead and that many of these elements including lead, which are common to both series, should have atomic weights varying according to their origin. Soddy and others have in fact, found that, whereas ordinary lead has an atomic weight varying from 206·98 to 207·22, uranium lead varies from 206·046 to 207·10, and thorium lead from 207·78 to 208·4. Such physical properties as density, solubility of salts, and spectra also vary, but, in the main, the two leads of atomic weight 206 and 208, as found in these various mixtures, must be as predicted on theoretical grounds, physically and chemically identical.

It is evident that atomic number, that is the number of the element in the periodic series, which is also the number of positive charges of the nucleus of the atom, and of orbital electrons around it, characterises an element chemically and physically, and that this may be different in two atoms of equal weight (isobares), or the same in two atoms of different weight (isotopes).

In the third chapter various attempts to fractionate known or supposed isotopic elements are described, and the reasons for failure in several cases are suggested. Repeated diffusion for 4 years, of hydrochloric acid gas through porous septa has yielded to Harkins and his fellow workers a gas containing chlorine of atomic weight 35·513 against the generally accepted value 35·46 and atomic weights of the constituents of 35 and 37. Kohlweiler has claimed to have obtained by diffusion an iodine of atomic weight diverging 0·66 per cent. from ordinary iodine. Unfortunately, Aston, on other grounds, has concluded that iodine is a simple element. Fractional distillation under low pressure has yielded Brönstead and Hevesy indecisive results for chlorine (hydrochloric acid was used), but marked differences for mercury.

Finally ,the methods of direct frontal attack on the atom itself are discussed. In all these methods the definite relation, e/m, of the electrical charge upon an atom to its mass is used to ascertain the latter. J. J. Thomson has done this in his method of positive ray analysis by studying the characteristic parabolas made on a photographic plate by positive rays propelled by an electric discharge in a rarified gas and deviated in their headlong course by an electrical and a magnetic field. Aston, by diverging the rays in an electrical and converging them in a magnetic field, has produced "mass spectra" in which each kind of atom or molecule present has recorded its ratio e/m in orderly fashion. Metallic elements seldom yield gaseous compounds suitable for positive ray analysis, but Dempster, G. P. Thomson and Aston have successfully utilised anode rays in obtaining, by means of the vital relationship e/m, records of the masses of the different atoms associated as isotopes in some metals. By these direct methods 19 elements, up to mercury (atomic number 80), have been shown to consist each of two or more kinds of atoms, tin, for example, containing a series of 7 or 8 atoms of weights from 116 to 124. In these 19 elements certainly over 60 different kinds of atoms are to be found. With the exception of hydrogen the atomic weights found by Aston are, within an approximation of $\frac{1}{1000}$, whole numbers. All this, and much more, is told in clear simple language; 157 references to original papers are given (more than one-third of these to English publications). The illustrations are mostly taken from these papers. Three-coloured diagrams, in which isotopes are also isochromes are used to make clear the inter-relations of the radio-active elements. The author, in most cases, both arrays in some detail the evidence submitted by workers for their views and keenly criticises its adequacy.

Of what interest is this to analysts? In the first place, the study of isotopes leaves the old elements in their places in the periodic table—that is, it confirms the feeling of all chemists that those elements are chemical individuals, and shows that this is so because the atoms of differing mass in an isotopic element are in the same electrical condition. Lead appears to be the one outstanding case where differences of origin have seriously affected the statistical identity of various samples found in nature. The atomic theory has been indicated, not only as the statistical basis of analytical chemistry, but, in its extended form, as a conception closely approaching reality, whilst the soundness of Prout's hypothesis has been shown. The exact experimental work expended on atomic weights and on the newer atomicity has yielded fruits which fully justify the statement of Plato: "If arithmetic, mensuration, and weighing be taken away from any art, that which remains will not be much." J. H. COSTE.

MANIPULATIONS DE CHIMIE COLLOIDALE. By W. OSTWALD. Translated from the 4th German Edition by E. VELLINGER. Pp. 202, 21 figs. Paris: Gauthier-Villars et Cie. 1924. Price 10 fr.

Ostwald's well-known "Kleines Praktikum" went through four editions in two years, and is recognised as an excellent manual on the practical side of Colloid Chemistry. Prof. Vellinger has made a faithful translation of this work into French. Thus, chemists, biologists, and others, able to read French better than German, will welcome this translation and find it thoroughly useful.

No fewer than 183 experiments, classed under various headings, are set out with detailed quantitative data, and they have the great merit of being guaranteed to "go." The selection of the experiments has been well made, considering the very wide field now included in colloid chemistry. Incidentally, the reviewer would prefer to see other methods included for determining surface tensions, and also a better account of the very useful flocculation-meter, in connection with which no mention is made of the work of F. V. von Hahn.

A notable omission is the very important subject of emulsions. Future editions should certainly include experiments on the making, breaking, and reversal of emulsions, and, possibly, some elementary experiments dealing with disperse systems in gases.

Colloid chemistry has many peculiarities in its methods and phenomena, and this book, an English edition of which is promised, provides a sound introduction to the practical side of a branch of science of daily-increasing importance.

W. CLAYTON.

CLOUDS AND SMOKES. By W. E. GIBBS, D.Sc. With Foreword by SIR OLIVER LODGE, F.R.S. Pp. 240. London: J. & A. Churchill. 1924. Price 10s. 6d. net.

In this book Dr. Gibbs has collected together from widely differing sources most of the available information on solids and liquids dispersed in gaseous media.

Recent work has shewn that certain types of aerosol are the counterpart in many respects of the better known and stabler hydrosols or colloidal solutions, and Dr. Gibbs has performed a very useful service in emphasising the colloid aspect of clouds and smokes and in presenting the material from the standpoint of the chemist. When, however, all has been said, our knowledge of this field, as the text shews, is surprisingly scanty, and, apart from the Brownian movement, the characteristics of these systems have scarcely been defined. Thus Chapter IV., which deals with the mechanical, thermal, optical, and electrical properties of aerosols, is suggestive but unsatisfying. Some of the deductions from first principles are unsupported by direct evidence, such as the statement on page 60 that inert dust may lower the ignition temperature of an explosive mixture of gases, or on page 61 that "the evaporation of liquid drops is arrested by the presence of an oil film."

Further, the evidence from many authors is accepted without any critical discussion of their conclusions, but this reflects the unsatisfactory state of our knowledge rather than any want of discrimination on the part of the author. The absence of definite information on the adsorption of gases by smoke particles is a case in point. Adsorption may be the main factor in determining the stability of many systems, but no quantitative measurements are available.

The mechanism of thermal precipitation, again, raises many interesting questions, but since Aitken's work little has been done. The intensity of light radiated from small particles is a subject in which fresh experimental work is needed. Bancroft has pointed out the disagreement between the work of Tolman on smokes and that of Mecklenberg on suspensions of sulphur. In most smokes the sizes of the particles lie well below the wave-length of sodium light and in a region where the two effects of reflection and scattering are difficult to disentangle.

The electrical character of smokes is very obscure. Undoubtedly, ionisation of the surrounding medium is largely responsible for the charges on the particles, but it is unknown whether smokes, as a whole, are electrically neutral, or whether preferential adsorption of ions takes place to any extent. Smokes produced by chemical reactions at low temperatures contain, initially at least, very few charged particles, but it is not known whether this proportion changes with time, and whether an equilibrium is reached between molecules, free ions, and adsorbed ions. In other words, is there a definite contact potential between a smoke particle and the surrounding medium?

These and many other questions are suggested on reading the first half of this book. It is clear that there is here a large field awaiting investigation, and Dr. Gibbs' treatment cannot fail to stimulate interest in this little explored region of disperse matter.

The second half of the book treats of the practical and industrial aspects of clouds and smokes. The subjects dealt with range over meteorological phenomena, smoke filtration and precipitation, dust explosions, and chemical warfare. A good account is given of the Lodge-Cottrell process and its applications, and the difficulty experienced in precipitating certain smokes, which the author ascribes to the reflection of ions, shews again how little the fundamental behaviour of aerosols is understood.

Dr. Gibbs is to be congratulated on a very complete review of the whole literature of the subject; copious references are given, and this monograph, which is the only work of its kind, will appeal both to the chemical engineer and the student of pure science. R. WHYTLAW GRAY.

MONOGRAPHS ON MINERAL RESOURCES, WITH SPECIAL REFERENCE TO THE BRITISH EMPIRE. Prepared under the direction of the Imperial Institute. VANADIUM ORES. London: John Murray. 1924. Price 5s. net.

The general preface explains the object of this series of monographs—"to give a general account of the occurrences and commercial utilisation of the more important minerals, particularly in the British Empire. No attempt has been made to give details of mining or metallurgical processes."

This little monograph gives a short historical account of vanadium, a description of the principal ores, their occurrence, the views held as to their genesis, and an indication as to where vanadium ores are being raised at the present time.

A commendable feature is that the volume contains a bibliography of the references used in the compilation of the monograph. This in itself has a marked value.

Amongst comprehensive references to sources of supply, the description of the famous deposit at Quisque, now the world's chief source of vanadium, which "has contributed about 80 per cent. of the world's production," has extreme interest; for the discovery, in 1905, that that which was formerly considered to be coal, was in reality an asphaltite associated with the mineral "patronite" (which contains vanadium as V_2S_5+4S) is one of the romances of modern mining.

It may have interest to add that, in addition to deposits mentioned in S. America—Argentine, Chile, and Peru, there is a further deposit at the extreme north, namely Venezuela, upon which the Reviewers did considerable work many years ago. The ash of this particular asphaltite carried 11·0 per cent. of V_2O_5 and 1·86 per cent. of NiO.

For the layman, the uses of the metal and a brief outline of its metallurgy are presented; for the student or professional worker, the material here compiled will be found to be reliable. As a general survey of vanadium deposits, the vicissitudes of their workings in various localities, and as some account of the causative factors for the same, this book fulfils a useful purpose.

GEO. R. THOMPSON.
JOHN MYERS.

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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The Routine Examination of Dairy Products with Special Reference to the Mojonnier Tester.

By L. H. LAMPITT, D.Sc., F.I.C., E. B. HUGHES, M.Sc., F.I.C., and M. BOGOD, B.Sc., A.I.C.

(*Read at the Meeting, April* 2, 1924.)

In any creamery or factory where dairy products are in process of manufacture, it often becomes necessary to make estimations of fat and solids-not-fat in short spaces of time if the results of the control are to be of any practical use. Such a case is to be found in the manufacture of ice-cream on a large scale, where every "mix" of, say, 300 gallons, has to be standardised before it can pass to the freezing machines.

We have found the Mojonnier tester to be most useful in such a case, and the following work is presented, not only to demonstrate the ease and speed of manipulation, but also to show the reliability of the results obtained by this method.

It is not proposed to describe the Tester, as a full description will be found in Mojonnier and Troy's book on the technical control of dairy products,* but it may be said that, in a comparatively small space it combines the means of making rapid and accurate estimations of the total solids and the fat in milk and its products, the total solids being estimated by drying in vacuo and the fat by a modified Röse-Gottlieb method. The economy in time effected by using this method will be appreciated when it is stated that it is possible by means of the Total Solids apparatus, to estimate the solids in milk in 21 minutes, whilst the time required for estimation in duplicate is only 30 minutes. For other dairy products varying times are required, as is shown in the following table:

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* Mojonnier and Troy, *Technical Control of Dairy Products*, 1st ed., pp. 62–69. (Published by Mojonnier Bros. Co., Milk Engineers, Chicago, 1922.)

TABLE I.

Quantities of materials used and the time required in the vacuum oven for estimation of total solids.

Product.	Milk.	Cream and Butter.	Dried Milk Products.	Ice Cream.	Cheese.	Evaporated Unsweetened Milks.	Sweetened Condensed Milks.
Wt. of material (grms.)	2	0·5–1·0	0·5	2	0·5	1·0–2·0	0·5
Time in oven (mins.)	10	10	15	10	20	15	20
Total time required for the estimation	21	21	26	21	31	26	31

With sweetened condensed milk there is always a certain amount of moisture remaining, and accordingly, when working under the conditions specified above, 0·3 per cent. is subtracted from the solids content, as it has been found that this is the additional loss after 90 minutes in the vacuum oven.

That the results obtained are consistent is shown in Table II., which gives typical results of duplicate estimations obtained in our laboratories with various products.

TABLE II.

Typical results of duplicate estimations of Total Solids.

	Sample I.		Sample II.		Sample III.		Sample IV.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Milk	12·22	12·24	11·36	11·35	12·21	12·23	12·30	12·33
Cream	55·65	55·61	39·52	39·60	54·45	54·45	54·84	54·79
Butter	84·46	84·47	84·31	84·28	85·78	85·80	84·46	84·51
Milk powder	97·82	97·80	97·75	97·70	98·22	98·24	98·02	97·99
Ice cream	38·06	38·03	34·59	34·63	34·51	34·53	34·95	34·95
Cheese	65·77	65·70	48·26	48·22	72·65	72·71	76·80	76·72
Unsweetened condensed milk	25·64	25·69	37·73	37·65	30·94	31·00	39·81	39·80
Sweetened condensed milk	76·11	76·21	73·50	73·33	78·47	78·36	72·90	72·96

It may also be stated that the apparatus is being used with success in the estimating of total solids in sauces, syrups, confectionery, tomato purée, fruit pulps, etc.

The Mojonnier modification of the Röse-Gottlieb method for the estimation of fat has been made particularly simple and rapid, the time occupied for a complete estimation being 30 minutes.

As with the total solids estimation, varying quantities of materials are required according to their type, as is shown in the following table:

TABLE III.

Quantities of Materials required for Fat Estimations.

	Grms.
Milk	10
Ice cream	5
Evaporated milk	5
Sweetened condensed milk	5
Thin cream	2
Rich cream	1
Butter, cheese, and other solid products such as milk powder, etc.	1

In nearly every case it is found that all the fat is removed in the two extractions specified in the Mojonnier method, and that a third extraction gives an additional weight of only 0·2 to 0·3 mgrm., which can be accounted for by the residue from the added solvents. We have found that the only case in which the whole of the fat is not obtained in 2 extractions is when working with spray process milk powder, which requires three extractions, no further increase in weight being obtained with additional extractions. Certain investigations were carried out to see if the ammonia used in the process had any saponifying action on the butter fat. For example, the residues after extraction were acidified and the mixture re-extracted with ether and petroleum spirit. It was found that in no case was the increased weight thus obtained more than could be accounted for by the traces of impurities in the solvents used, showing that no saponification had taken place. On re-extracting the fat with petroleum spirit in the dishes, the loss in weight of each dish was exactly the weight of the fat previously found.

This is also borne out by the figures given in the following table, which shows the results of complete estimations carried out on weighed quantities of butter fat:

TABLE IV.

Butter Fat Extractions.

Butter fat taken. Grms.	Butter fat found. Grms.
0·1362	0·1362
0·2100	0·2097
0·3463	0·3465
0·5192	0·5188
0·5638	0·5641

A point which we have found to be of major importance is the amount of shaking given to the contents of the extraction flasks, and unless this is carried out vigorously and for the space of time specified, abnormal results will be obtained, as is shown in the following table, where comparative sets of figures are given for thorough mixing and also for continually inverting the flask for the same period. (Table V.)

TABLE V.

Effect of Shaking on the Estimation of Fat.

Material.	Vigorous shaking.		Gentle shaking.	
	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Milk powder	26·07	26·06	24·55	25·32
Milk powder	26·34	26·31	24·62	24·80
Cream	49·54	49·50	48·81	49·05
Cream	50·62	50·69	49·62	49·93

In order to make the conditions of the extraction more comparable with those obtaining during the analysis of a milk product, estimations were made on a mixture of butter-fat and skim milk powder (fat content 1·51 per cent.), the weights of each substance used being approximately those of fat and solids-not-fat in 1 grm. of cream containing 50 per cent. of fat. The results obtained are shown in the following table, together with those obtained by other methods, the comparative tests being made to show relative results which would be obtained in the ordinary course of analysis, and not as a detailed investigation on the accuracy of the other methods. (Table VI.)

TABLE VI.

Estimations of Fat in mixtures of butter-fat and skim-milk powder.

(Skim-milk powder used contains 1·5 per cent. butter-fat.)

Method.	Total weight of Fat. Grms.	Weight obtained. Grms.	Difference as per cent. of fat taken.
Babcock. (Reading to bottom of meniscus × 0·18)	4·933	4·986	+1·07
	4·198	4·284	+2·03
Gerber. (One c.c. amyl alcohol used. Reading to bottom of meniscus × 0·111 and 0·0054 added for meniscus	0·495	0·504	+1·82
	0·502	0·509	+1·40
Gerber. (Previous reading × 0·110 and 0·0054 added for meniscus.)	0·495	0·499	+0·81
	0·502	0·505	+0·60
Gerber. (0·25 c.c. amyl alcohol used. Reading × 0·111 and 0·0054 added for meniscus.)	0·519	0·519	0·00
	0·496	0·494	−0·04
Mojonnier. (Two extractions.)	0·5297	0·5298	+0·01
	0·5080	0·5075	−0·10
Röse-Gottlieb. (Three extractions.)	0·2577	0·2580	+0·12
	0·2835	0·2835	0·00
Werner-Schmid. (Three extractions.)	0·5106	0·5133	+0·53
	0·6097	0·6127	+0·49

Details of the estimations recorded in Table VI. were as follows:

(*a*) The Babcock method was carried out with the constituents of 9 grms. of cream; the reading from the base of the fat column to the bottom of the upper meniscus being multiplied by 0·18 to obtain the weight of fat represented by the reading.

(*b*) In the Röse-Gottlieb estimation the equivalent of 0·5 grm. of cream was used.

(*c*) The Gerber results were arrived at in three different ways, depending on various quantities of amyl alcohol used, and also on two types of calculation.

(i.) Using 1 c.c. of amyl alcohol. Reading from base of the fat column to the bottom of the upper meniscus multiplied by 0·111, and 0·0054 added. (Based on Day and Grimes' results.*)

(ii.) Using 1 c.c. of amyl alcohol. Reading multiplied by 0·110 and 0·0054 added.

(iii.) Using 0·25 c.c. of amyl alcohol. Calculation as (i.).

From these results it appears that the recovery of fat is most nearly the theoretical amount in the modified Gerber, the Mojonnier, and the Röse-Gottlieb methods.

The whole series of methods was extended to different milk products, with the results shown in Table VII.

TABLE VII.

Typical estimations of fat.

	Babcock. Per Cent.	Gerber I.† 1 c.c. amyl alcohol. Per Cent.	Gerber II.† 0·25 c.c. amyl alcohol. Per Cent.	Röse-Gottlieb. Per Cent.	Werner Schmid. Per Cent.	Mojonnier. Per Cent.
Milk	3·35	3·40	—	3·37	3·45	3·40
	3·35	3·40	—	3·37	3·45	3·40
Cream	48·8	48·3	48·2	48·16	48·36	48·23
	48·8	48·3	48·2	48·27	48·23	48·27
Cheese	34·8	38·8	35·3	35·45§	35·63	35·50
	34·8	36·8	35·3	35·45	35·69	35·54
Milk powder (full cream), roller process	24·8	25·4	—	25·48	25·50	25·50
	24·2	25·4	—	25·40	25·51	25·54
Milk powder (full cream), spray process	25·0	27·5	—	27·26	27·01	27·35
	25·4	27·5	—	27·30	27·12	27·37

In connection with these figures the following points should be noted:

(*a*) In the Gerber, Mojonnier and Werner-Schmid methods, 1 grm. of material was weighed out in all cases except for milk.

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* ANALYST, 1893, **18**, 123.

† Gerber I. Factor: Reading × 0·110 + 0·0054; Gerber II. Factor: Reading × 0·111 + 0·0054.

§ Double the strength of ammonia.

(*b*) In the Babcock method 2 grms. were weighed in the milk-bottle; for cream 9 grms. were weighed out, and a cream bottle used.

(*c*) In the ordinary Röse-Gottlieb estimation 0·5 grm. of material was used.

(*d*) In the case of spray process powder, the material was weighed into a small beaker, dissolved in water, and poured into the vessel in which the estimation was to be made, the beakers being subsequently rinsed with the reagents used.

These results bear out the conclusion drawn from the results given in the previous table.

A point of great importance, and one which we do not think has been sufficiently recognised, is the effect of the quantity of amyl alcohol on the reading of fat in the case of estimations by the Gerber method in cream.

If only 0·25 c.c. of alcohol is used, a correct result is obtained by using the factor 0·111 and adding 0·0054 for the meniscus, but if 1 c.c. of alcohol is used, abnormally high figures result, as the fat column contains an appreciable quantity of amyl alcohol and amyl esters. Actual analysis of such fat showed the presence of 2·28 per cent. of volatile matter, of which 1·44 per cent. was amyl alcohol and 0·78 per cent. amyl esters, calculated as amyl butyrate.

Results show that the Babcock method is not very accurate, and this has been pointed out by American workers.*

The great difficulties which have been noted by various workers in the estimation of fat in spray process milk powder, and which we ourselves have experienced in the course of extended studies on such powders, have led us to test many methods. In general, as a routine method we have adopted the Gerber method, using a weighed quantity of powder as described above, and whirling two or three times for 3 minutes at about 1300 revs. per min. till the reading is constant. We find that the value of 0·110 grm. of fat for each division and an allowance of 0·0054 grm. for the meniscus gives satisfactory results, readings being made at 65 °C.

That the Mojonnier modified Röse-Gottlieb method gives consistently reliable results is shown by the following typical estimations. (Table VIII.)

TABLE VIII.

Typical duplicate estimations by means of the Mojonnier Tester.

Product.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.	Per Cent.
Milk	3·10	3·10	3·40	3·40	3·72	3·73	3·52	3·52
Cream	49·58	49·60	51·30	51·24	50·72	50·74	48·97	49·00
Cheese	35·50	35·54	27·18	27·18	34·96	34·89	31·51	31·55
Unsweetened condensed milk	8·99	8·98	9·54	9·54	10·36	10·34	9·12	9·13
Milk powder	27·00	27·02	26·78	26·75	26·96	26·97	27·10	27·11
Ice cream	13·94	13·95	14·22	14·23	14·26	14·26	14·32	14·31
Sweetened condensed milk	8·53	8·53	9·02	9·01	8·74	8·76	9·38	9·35

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* Mojonnier, *Technical Control of Dairy Products*, p. 51; *Dairy Science*, VI., p. 549; and *J. Ass. Off. Agr. Chem.*, 7, 159 *et seq.*

Our results may be summarised as follows:

(1) The total solids of milk products can be estimated rapidly and with consistent results by means of the Mojonnier tester. A single estimation can be made in 21 minutes; in duplicate in 30 minutes.

(2) The Mojonnier modification of the Röse-Gottlieb method for the estimation of fat has been shown to give accurate results. In the case of spray process milk powder three extractions are recommended.

(3) The time required for one estimation is 30 minutes. Both fat and total solids can be estimated in 35 minutes.

(4) A modified Gerber method is recommended for the routine estimation of fat in milk powders.

(5) It has been shown that where the proportion of fat to the milk solids is high, as in cream, the amount of amyl alcohol used in the Gerber method affects the result to a considerable extent.

We wish to acknowledge with thanks the permission of Messrs. J. Lyons and Co., Ltd., to publish these results.

Discussion.

Dr. L. H. Lampitt said that, originally, their reason for taking up the Mojonnier apparatus was a purely commercial one. When in America he had met and been rather impressed by Dr. Timothy Mojonnier—who was a chemist first and a laboratory and dairy-equipment expert second—and had inspected many dairies and creameries in which the Mojonnier equipment was used. At that time his firm had been unable to compete in their laboratory with the ice-cream question. They had a plant with a capacity of 25,000 gallons of ice-cream per day; this was divided over 600 gallon mixes during 20 hours, which gave them 45 minutes to analyse samples. The Mojonnier Tester afforded an excellent method of obtaining the necessary control, and, in his opinion, it was as good as any other routine method of determination, its great advantages being accuracy and speed. It was certainly an expensive apparatus, but it had proved itself to be well worth its cost. Each test cost 8½d. in materials, but the expense was balanced by the speed with which the tests could be carried out. His firm proposed to instal another Mojonnier tester to be used entirely for moisture estimations in the Baking Section. It was anticipated that this apparatus would turn out to be as efficient as that used for ice-cream.

Mr. Hinks enquired whether the percentage of total solids of milk obtained in the Mojonnier tester was the same as that obtained by drying the milk at 100° C. for three hours in a water oven in the usual way.

Mr. F. E. Day said that in his experience he had found the Mojonnier apparatus useful for estimations of fat, but had always felt dubious about evaporating in open pans with ether. For moisture they had had good results with new milks, but with condensed milks they had obtained very curious results—after 10 to 20 minutes' drying they had obtained a figure which corresponded to the water of hydration of milk sugar.

Mr. E. R. Bolton, remarking upon the great necessity for speed which had mainly prompted the authors to instal the Mojonnier apparatus, made the suggestion that the time might be still further curtailed by a modification of the procedure on the lines of the process suggested by Revis (Analyst, 1907, **32**, 284), in which a small quantity of acetone was added before evaporation of the milk in estimating total solids. He always used this method, not only for milk, but also for condensed milk, and its rapidity and accuracy were astonishing. In one case he had carried out a series of tests which were duplicated with the Mojonnier apparatus in America, and the results agreed as closely as if they had been carried out by the same observer in one laboratory.

Mr. G. **Rudd Thompson** said that he could see no reason why results obtained in this way should not be equal to those obtained by the ordinary laboratory methods; they ought to be better. In his opinion, the evaporation of an ethereal solution of fat-solids under glass entailed a great waste of material (he referred more especially to the ether carried through a fan to the open air) it was not only a great waste, but there was a danger from fire.

The Freezing Point of Sudan Milk.

By A. F. JOSEPH, D.Sc., F.I.C., and F. J. MARTIN, M.A., Ph.D., F.I.C.

The recent improvements in apparatus for determining the freezing point of milk have led us to use the method for obtaining additional information on which to base a comparison between the properties of milk of tropical and temperate climates. In a previous communication(1) we drew attention to the fact that cows' milk in the tropics is usually richer both in fat and solids-not-fat than that in temperate climates, and we are now able to supplement this by showing that in the Sudan cows' milk has the same freezing point as has been reported elsewhere. Some data have also been collected relating to the milk of other animals.

In carrying out this work we have used the cryoscope described by Dr. Hortvet in 1921(2); the method was critically examined by the Association of Official Agricultural Chemists and reports were published in 1922 and 1923.(3, 4) A few years before Hortvet's work appeared, the question was investigated by Monier-Williams,(5) who, however, considered that the experimental difficulties involved in obtaining reliable results were such that the method was not likely to be adopted. Using Hortvet's cryoscope and following his instructions, we have obtained very concordant results and are satisfied that no special skill is required in its use.

The Lowering of the Freezing Point of Cows' Milk.—The first point investigated was the effect of the breed of animal on this property. For this purpose samples were taken from 51 animals of three different breeds, with the following results:

Breed.		Demietta.	Shorthorn crosses.	Native Khartoum district.
Number examined		16	9	26
Freezing-point depression, °C.	Average	0·558	0·553	0·556
	Highest	0·579	0·578	0·573
	Lowest	0·538	0·538	0·546
Fat, per cent.	Average	3·8	4·4	4·1
	Highest	5·8	5·7	5·6
	Lowest	2·5	3·6	3·3
Solids-not-fat, per cent.	Average	9·0	9·3	9·1
	Highest	9·6	10·0	10·0
	Lowest	8·7	8·7	8·6

The average lowering of freezing point for all the samples is 0·556, that for the different breeds being substantially the same. The mean value for 129 results quoted by Bailey(3) is 0·548, the limits being 0·530 to 0·566. None of our samples has given a lower value than 0·530, and only four were higher than 0·566. As a difference of ·01 °C. in the freezing point corresponds to only 2 per cent. of added water, we should be justified in using the standard recommended for America.

Variation in Depression of Freezing Point in Different Portions of the same Milking.—A common difficulty in dealing with the milk of individual animals is caused by the great difference in fat content of the first and later portions of the milking. The following results show that the freezing point is practically the same throughout.

Percentage of Fat.

Cow No.	1st portion.	2nd portion.	3rd portion.	4th portion.
70	2·5	3·7	3·0	5·1
73	1·3	1·8	2·9	6·9
18	2·2	3·5	4·7	6·2
75	1·8	2·4	1·7	5·7
Mean	1·95	2·85	3·07	5·97

Freezing Point Depression.

	°C.	°C.	°C.	°C.
70	0·561	0·558	0·559	0·559
73	0·561	0·559	0·562	0·559
18	0·570	0·569	0·568	0·568
75	0·556	0·556	0·558	0·553
Mean	0·562	0·560	0·562	0·560

Variation of Freezing Point Depression with the Period of Lactation.—The samples from which the results were given in the first table were taken from animals at various stages of their lactation period, and, when grouped according to this, give the following results:

Period of Lactation.	No. of Animals.	Average Depression of Freezing Point. °C.
1st Month	8	0·552
2nd „	12	0·560
3rd „	8	0·556
4–6th „	10	0·553
7–10th „	14	0·554

These differences are unimportant.

Depression of Freezing Point of the Milk of Animals other than Cows.—A number of samples from goats, sheep, and asses, and one of camel's milk, gave the following results:

Animal.		Goats.	Sheep.	Asses.	Camel.
Number examined		12	8	6	1
Freezing-point depression, °C.	Average	0·575	0·564	0·554	0·616
	Highest	0·591	0·591	0·563	—
	Lowest	0·564	0·547	0·546	—
Fat, per cent.	Average	4·1	6·1	0·7	4·7
	Highest	7·5	8·2	1·9	—
	Lowest	2·8	3·0	0·1	—
Solids-not-fat, per cent.	Average	9·2	11·7	7·6	8·6
	Highest	10·3	12·5	8·5	—
	Lowest	8·8	10·9	5·4	—

In the case of 4 goats, the milk was drawn in two portions which were examined separately.

	1st Portion.			2nd Portion.		
Goat No.	Depression F. Point. °C.	Fat. Per cent.	Solids-not-fat. Per Cent.	Depression F. point. °C.	Fat. Per Cent.	Solids-not-fat. Per Cent.
1	0·570	2·8	8·5	0·570	3·6	8·5
2	0·591	3·3	9·7	0·593	5·3	9·3
3	0·570	4·0	9·3	0·570	5·2	9·3
4	0·570	3·2	9·3	0·571	3·9	8·8
Mean	0·575	3·3	9·2	0·576	4·5	9·0

FACTORS WHICH INFLUENCE THE FREEZING POINT DEPRESSION.—In the reference already quoted(3, 4) attention is drawn to certain conditions which may cause abnormal freezing points in genuine samples. Bailey finds that the morning milk has a larger depression than the evening, but the differences seldom exceed 0·02° C., and are frequently less than ·01° C. We have not, up to the present, collected any local data on this point, but the differences are not great enough to impair seriously the usefulness of the method.

The case of diseased animals always presents difficulty, but, for practical purposes, they might well be grouped as "non-genuine."

A common cause of an increased depression of freezing point is the development of acidity. Bailey has devoted much attention to this point and confirms the opinion of Monier-Williams, that where the sample does not smell and taste distinctly sour, the freezing point is unlikely to be depressed by as much as 0·002° C.

In hot weather here the acidity increases so rapidly that a preservative must be added if the examination is delayed even for only a few hours. We have found mercuric chloride, added in the proportion of 1 in 2000, to be very satisfactory, and this is illustrated by the following results for a sample tested immediately on receipt and after standing 24 hours, with and without the addition of mercuric chloride:

	Depression of freezing point.	Acidity (i.e. c.c. of 0·02 *N* alkali required per litre of milk).
On receipt	0·578	18
After 24 hours	0·806	95
Do., but $HgCl_2$ added	0·578	20

SUMMARY.—1. The freezing point of cows' milk in the Sudan is practically the same as in the United States.

2. Different portions of the same milking give the same freezing point.

3. The period of lactation only exerts a very small influence on freezing point.

4. The milk of goats and sheep gives a greater depression of freezing point than that of cows; that of asses gives the same.

5. Abnormal results due to the rapid development of acidity in a hot climate are prevented by the use of mercuric chloride (one part in 2000).

Our thanks are due to the Director of the Veterinary Department and to the staff of the Sanitary Service, Khartoum, for furnishing us with the samples.

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(4) Do. Vol. VI., page 422.
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Alcoholysis and the Composition of Oils and Fats.

BY G. D. ELSDON, B.Sc., F.I.C.

IT is indeed refreshing to read the remarks by Channon, Drummond and Golding in a recent paper in the ANALYST (1924, 49, 311) in connection with the determination of the composition of the mixed fatty acids of fats by the method of fractional distillation of the methyl or ethyl esters. Already, in 1913, the present writer (ANALYST, 1913, 38, 8), as a result of a certain amount of work with this process, stated:—"From the experiments carried out it has been concluded that the process of alcoholysis is a suitable one for determining the qualitative composition of an oil, and that it also gives considerable information in regard to the quantities of the constituents present; the process, however, is much too lengthy for use as an ordinary laboratory test, and would seem to be chiefly of theoretical interest. The quantitative results have not been encouraging, but the process probably gives results within about 5 to 10 per cent. of the true value—that is to say, it should decide between 35 and 45 per cent. for the content of lauric acid in coconut oil," and this opinion was repeated in a later paper (ANALYST, 1914, 39, 78), although in the latter paper, unfortunately, there is a fairly obvious misprint in the last line but one, which possibly somewhat obscures the meaning.

Of late years, this method has been very largely used both in this country and in America for the elucidation of the composition of oils and fats, and some extraordinary claims have been made for the process. These claims are, in the opinion of the writer, not justified by the facts and a short time ago the following sentence was written by him (*Chemical Age*, **1924**, **10**, 188): "It is perhaps a little difficult to understand why the only one of the acids of high boiling point which appears in the lower fractions should be oleic, and also, to take one example, why in one of the lower fractions there should be 0·608 grm. of caproate, 0·320 grm. of caprylate and 0·160 grm. of oleate to the total exclusion of all other esters. The experience of the writer in the fractional distillation of large quantities of methyl esters obtained from coconut oil would, moreover, scarcely support this assumption. . . ." With all these ideas Channon, Drummond and Goulding are evidently in perfect agreement, and they give a most excellent account of their objections in their recent paper quoted above, the extraordinary and inexplicable excellence of the results obtained by Crowther and Hind on their artificial mixture of fatty acids notwithstanding. They do not, however, draw particular attention to the enormous differences in composition found by Crowther and Hynd and by Holland and Buckley, respectively, in their examination of butter fat. The largest amount of stearic acid found by the former was 5·9 per cent., whilst the figures obtained by the latter varied from 7·8 to 20·4 per cent.

The different results which may be found by various workers with the method of alcoholysis, and other methods, are well shown by the variation in the figures which have been obtained on the composition of the mixed fatty acids of coconut oil.

It has been shown by Jensen (ANALYST, **1905**, **30**, 397) that coconut oil contains no butyric acid and but little caproic, the volatile acids being mainly capric and caprylic. Paulmyer (*J. Soc. Chem. Ind.*, **1907**, **26**, 881) found that the fatty acids of coconut oil consisted of capric, 20 per cent.; lauric, 40 per cent.; myristic, 25 per cent.; palmitic, 11 per cent.; and oleic, 5 per cent.; together with capric and caprylic acids; while Caldwell and Hartley (ANALYST, **1909**, **34**, 274) found by a different method at least 30 per cent. of lauric acid. Haller and Youssoufian found, by the method of alcoholysis, caproic, caprylic, capric, lauric, myristic, palmitic, stearic, and oleic acids. They did not give any quantitative results, but they stated that lauric and myristic acids predominated, whilst palmitic, stearic, and oleic were present in relatively small quantities. The writer, also, by the method of alcoholysis, deduced the following composition:

Caproic acid	2 per cent.
Caprylic acid	9 ,, ,,
Capric acid	10 ,, ,,
Lauric acid	45 ,, ,,
Myristic acid	20 ,, ,,
Palmitic acid	7 ,, ,,
Stearic acid	5 ,, ,,
Oleic acid	2 ,, ,,
	100 per cent.

but in this case the quantities of capric acid and stearic acid are possibly too high, whilst that of oleic acid is probably too low. Walker (*J. Chem. Soc.*, **1923**, **123**, 2837) has shown that, in a sample of coconut oil examined by him by a method of fractional precipitation, it was unlikely that more than 2 per cent. of capric acid was present. In the most recently quoted examination of coconut oil, referred to by E. F. Armstrong and John Allen in the recent Presidential address to the Society of Chemical Industry (*J. Soc. Chem. Ind.*, **1924**, **43**, 207 T), the following figures are given, which figures are stated to have been obtained by the method of alcoholysis:

Caproic acid	0·25	per cent.
Caprylic acid	0·25	„ „
Capric acid	19·5	„ „
Lauric acid	40	„ „
Myristic acid	24	„ „
Palmitic acid	10·6	„ „
Stearic acid	0·0	„ „
Oleic acid	5·4	„ „
	100·0	per cent.

The extraordinary differences between these figures and other figures obtained both by the same and other processes, all of which differ among themselves, can, presumably, mean only one of two things, or possibly a mixture of both—either that there are enormous differences in the composition of coconut oils from different sources, or that, as is alleged by Channon, Drummond and Golding, the fractionation of the esters as an exact quantitative method is of little value.

In regard to this first point, there are undoubtedly differences in the composition of different samples of coconut oil, but the wide fluctuations in the composition, as indicated by the so-called quantitative analyses, are by no means upheld by the somewhat narrow variations in the analytical figures usually obtained. In the opinion of the writer, as has been previously expressed, the second is by far the more important of the two reasons, and, although the method of alcoholysis is undoubtedly valuable as a means of detecting the constituents of a mixture of fatty acids, it is only valuable in so far that better methods are, in general, not available.

The following remark, taken from Myddleton and Barry's recent book "Fats; Natural and Synthetic," shows the kind of confidence this method inspires, even in those who make use of it, "The figures indicated by the letter *a* in Tables VI.-IX. (*i.e.* tables which give the results of analyses of oils obtained in part by alcoholysis) have been adjusted by not more than 1·5 per cent.* of the observed figure to indicate clearly that no change takes place in the amount of this constituent, the 1·5 per cent. being within the limits of experimental error."

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* Whether this means 1·5 per cent. of the oil or 1·5 per cent. of the actual observed figure for the fatty acid is not too clearly stated—if the latter, then it may mean in some cases possibly ten per cent. or more of the oil.

The process may have some quantitative value, but for exact work it is not only useless but worse than useless, as it seems to be leading very seriously astray workers whose time might be better spent. The work of Channon, Drummond, and Golding in exposing the pitfalls into which some of the later workers seem to have fallen will be thankfully received by all those who have any desire to see the examination of fats placed on a sure footing. The present writer would take the liberty of emphasising everything that these authors have said, and would like particularly to draw attention to the remarks made by Dr. Drummond at the close of the discussion.

Note.

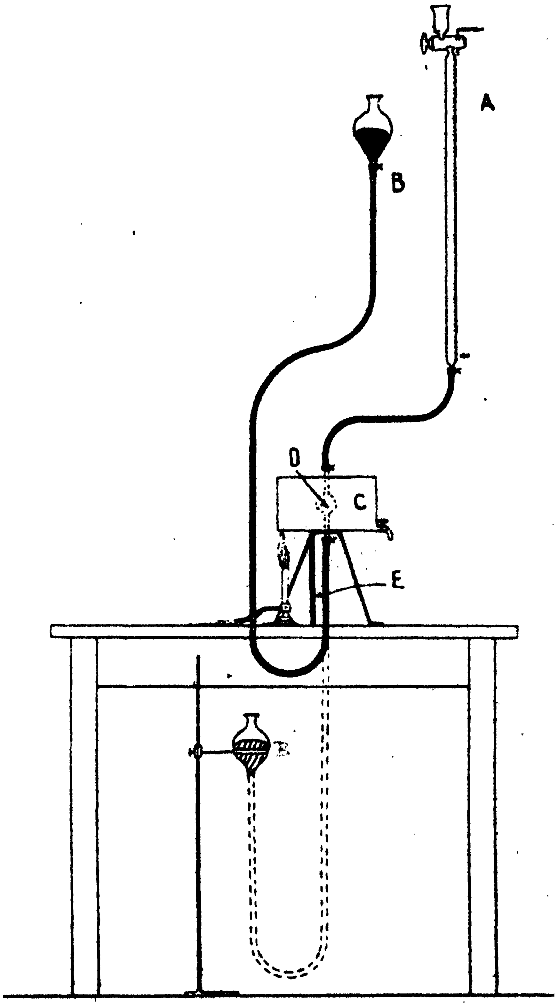
The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE ESTIMATION OF CARBONATES IN HIGHLY COLOURED LIQUIDS.

In the apparatus shown in the diagram A is a nitrometer connected at the lower end with a glass bulb, D, having a capacity of about 100 c.c. This bulb is also connected by pressure tubing with the mercury reservoir, B, which can be placed either above the working bench or below it in the position B′. The apparatus is filled with clean mercury. The bulb is set in an oval-shaped bath which can be heated at one side and emptied when necessary. The rubber tubing is protected from the flame by a screen, E.

The estimation is made as follows:—Twenty-five c.c. of the liquor are poured into the cup of the nitrometer. The liquor is drawn into the nitrometer by lowering B and opening the threeway cock. No air must be allowed to enter. The cup is washed with two separate quantities of about 10 c.c. of distilled water and both lots of wash water are drawn into the nitrometer. Twenty c.c. of 16 per cent. sulphuric acid are added and then 2 c.c. of "paroleine." The latter is to prevent the carbon dioxide which is evolved from dissolving in the residual liquor when the apparatus is cooling down. The mercury reservoir is lowered to the position B′, and the height adjusted so that all the liquor is in the bulb. The rubber connection between D and A is 16 to 18 inches long and, when an estimation is in progress, should be in a continuous upward sweep. When all the liquor is in the bulb, D, water at 65 to 70° C. is poured into the bath C, and the temperature maintained at that point. It is not advisable to overheat the bath. The liquor in D boils gently and is allowed to do so for 15 minutes. The liquor is then transferred to A by raising the reservoir B, and the whole is left to cool. The level of the mercury in B is maintained somewhat below that in A. The bath, C, is emptied.

The volume of the carbon dioxide evolved is measured, after cooling, by adjusting the height of the reservoir B in the usual manner. The observed volume should be reduced by 0·1 c.c. for every 10 c.c. of liquor, washings and acid. This represents the dissolved air and gives a sufficiently accurate correction for most purposes. (The actual value may be found after reading off the volume of the gas by running strong sodium hydroxide solution into the nitrometer, shaking well and then levelling up.)



In order to calculate the weight of carbonate as grms. of Na_2CO_3 per 100 c.c. of liquor, the formula (a) is used.

$$\frac{K\,(V-v)\,(P-B-c)}{(273+T)}\ \text{grms.} \qquad \text{(a).}$$

Here V represents the volume in c.c. of gas after levelling; v, the correction in c.c. for dissolved air; P, observed barometric pressure; B, pressure of aqueous vapour at T° C.; c, correction for temperature of mercury column; T, temperature of the laboratory in °C.; and K, = 0·006846; Log K = 3·8355.

The full formula is:

$$\frac{(V-v)\,(P-B-c)}{(273+T)}\left[\frac{273}{760}\times\frac{1\cdot977}{1000}\times\frac{106}{44}\times\frac{100}{25}\right]\ \ldots\ldots\ldots(b).$$

1·977 = weight in grms. of one litre of carbon dioxide at N.T.P.

106 = weight in grms. of sodium carbonate equivalent to 44 grms. of carbon dioxide.

The portion of formula (b) in square brackets = K.

The actual time required for this estimation (excluding the time occupied in boiling and cooling) is about half an hour.

HORACE BARRATT DUNNICLIFF.

THE CHEMICAL LABORATORY,
GOVERNMENT COLLEGE, LAHORE, INDIA.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

METROPOLITAN BOROUGH OF HAMMERSMITH.

ANNUAL REPORT OF THE PUBLIC ANALYST FOR 1923.

DURING the twelve months 626 samples were submitted for examination, of which 600 were purchased under the provisions of the Sale of Food and Drugs Acts, and 12 under the Public Health (Milk and Cream) Regulations. Of the total number examined, 509 were reported as genuine, 37 as adulterated, and 54 as of inferior quality. The percentage of adulteration was 6·1, as compared with 6·6 in 1922.

MILK.—Of the 435 samples examined, 54 were reported as inferior and 22 as adulterated. Four milks were found to be artificially coloured with annatto.

COCOA AND CHOCOLATE.—Four samples of cocoa, out of 25 examined, contained arsenic in quantities ranging from 1/130th to 1/80th of a grain of arsenious oxide per lb. Cautionary letters were sent to the vendors.

MEAT AND FISH PREPARATIONS.—Seven samples were examined, and, of these, 3 contained boron preservatives, the amounts found being 18·2 grains, 17·5 grains, and 13·3 grains (in terms of crystallised boric acid) per lb.

DRUGS.—Thirty-two samples were examined, and two of these (borax) were found to contain arsenic in the proportion of 10 parts of arsenious oxide per million. Cautionary letters were sent.

P. A. ELLIS RICHARDS.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

RICE HUSKS IN SHARPS.

ON May 17th, a firm of millers was summoned before the Norton (Malton) Bench of Magistrates for selling sharps which contained rice husks.

Mr. Hodge, prosecuting on behalf of the Ministry of Agriculture, stated that, on December 15th, 1923, the official sampler to the East Riding County Council had taken a sample of the sharps, but the defendants, who had been notified, did not attend. The sample was sent to Dr. Voelcker for analysis, and he reported that it contained some finely-ground rice husks, which were of no value as food, and action was accordingly taken under Section 6 of the Fertilisers and Feeding Stuffs Act.

On the back of the invoice was a printed notice to the effect that "all goods herein invoiced are, or may be, mixtures." This statement, counsel contended, was contrary to the spirit of the Act, and rendered void the special purpose for which that section was passed, i.e. to prevent a purchaser receiving any ingredient worthless for feeding purposes.

Dr. J. A. Voelcker, giving evidence in support of his certificate, said that a casual examination of the sample did not show anything wrong, but the microscope indicated the presence of rice husks. He had accordingly reported that the sample was impure, and that it contained a material worthless for feeding purposes.

Counsel for the defence urged that the question at issue was one of law rather than of fact. Defendants from the first admitted that, owing to the difficulty of obtaining wheaten sharps. it was necessary to add some ingredient. It was not suggested by the prosecution that rice husks were deleterious to poultry or stock, but only that they were worthless for feeding purposes. The question was whether they were worthless altogether. Rice husks had a value, and cost £5 9s. 8d. per ton at Hull, so that it would appear they had a value and were not, therefore, a worthless ingredient. On that ground he submitted that the summons would have to be dismissed.

A member of the defendant firm gave evidence to the effect that it was the custom in the milling trade to mix sharps, as in this case, but these were always sold as "mixed," and not pure wheaten sharps. They believed that rice husks had a food value, but when they were mixed with the sharps they charged less for the mixture.

The Chairman said the Bench had come to the conclusion that there could be no doubt that there had been adulteration, and agriculturists ought to be protected against that sort of thing. If they bought sharps they should receive sharps. As this was the first case of the kind to come before the Bench, they had decided to deal leniently with the four defendants, and a fine of £5 each, together with costs—£3 each—would be imposed.

APPEAL CASE UNDER THE RAG FLOCK ACT.

CHADWICK *v.* KERSHAW.

ON June 30th an appeal was made to the Bucks Quarter Sessions at Aylesbury by Messrs. H. E. Kershaw, Ltd., who, on May 29th had been fined £10 by the Wycombe Borough Magistrates for selling flock which did not conform to the standard of cleanliness prescribed by the Rag Flock Regulation of 1912. The sample taken contained 243 parts of soluble chlorine per 100,000, as compared with the permissible amount of 30 parts per 100,000.

Mr. Fox Davies, for the Borough Council, said that what the Court below had found was that the sample taken was not up to the Government standard of cleanliness, and that there were in it jute, coconut fibre, and pieces of woven material. If the sample had been all coconut fibre, no matter what its state of cleanliness, it did not come under the Act, but what the respondent maintained was that as woven material was present, it came under the Act, and it was on that ground that the Court below had convicted. The defence, he understood, was that the jute fibre was fed into the machine on pieces of Hessian cloth, and their contention was that it was not rag. He would contend, however, that if only a small portion of woven material were found, then it became rag flock and came under the Act.

Mr. A. P. Davson, F.I.C., said that he had analysed a sample of the material and found it to contain 243·0 parts per 100,000 of soluble chlorine. In that sample he had found about 8 per cent. of pieces of material which had evidently been woven. He had analysed coconut fibre, and his average results showed 70 parts of chlorine per 100,000. The composition of the sample might fairly be taken as: Coir fibre, 19 per cent.; pieces of spun and woven material, 8 per cent.; and jute fibre, 73 per cent. He came to the conclusion that the remainder of the jute material was probably woven, because the pointed ends which were common in raw jute could not be detected.

He was asked in cross-examination whether he was aware that coir fibre was steeped in tidal waters for a year or 18 months, and that this would account for the salt present in it, and that in this coir there was as much as 640 parts of chlorine per 100,000. Mr. Davson replied that neither his analysis nor that of others supported this assumption.

Several flock manufacturers gave evidence to the effect that they had no hesitation in saying that the material in question had been made out of old bagging, or sacking. It had been "pulled" in the ordinary way, but was unwashed. Commercially it was not possible to make flock out of fibre.

Mr. C. A. Mitchell said that he had analysed two samples of coconut fibre—one prepared at Mitcham and the other imported from Ceylon. These contained 14 and 19 parts of soluble chlorine per 100,000 respectively. He had also analysed the actual coir fibre in a sample of the mixture sold by the defendants, and had found it to contain 17 parts of chlorine per 100,000. Hence, it could not have been the source of the excessive chlorine in the material.

A manufacturer of coir fibre stated that the average annual amount of coir fibre imported from India was 300 tons, whilst that from Ceylon was 11,000 tons per annum. Ceylon fibre was not treated in salt water, but in mountain streams. Witness added that he was only giving evidence because an unmerited slur had been cast on coconut fibre, which, if not contradicted, would be injurious to the reputation of the fibre used for bedding and upholstery purposes.

Mr. Robertson, for the defence, stated that the only woven material present in this material was the long strip of washed Hessian cloth, which was used as a carrier for introducing the said jute fibre into the machine, and he called the defendants to give evidence to this effect.

The foreman at the works, also called, said that the material consisted of 50 per cent. of coir fibre, 45 per cent. of flax and hemp waste, and 5 per cent. of jute.

Dr. J. F. Shaw, A.R.C.S., called for the defence, said that he was very familiar with jute and that the sample produced was composed largely of raw jute fibre and coir fibre, with a few small strings derived from the Hessian cloth. He had not detected any trace of flax. He was familiar with the methods of preparing coir fibre in Madras, and understood that a satisfactory condition could not be obtained except in tidal waters.

Witness was proceeding to give text book figures as to the amount of chlorine in Indian coir, when exception was taken to this evidence by Mr. Fox Davies.

Mr. Fox Davies maintained that he had proved beyond doubt the same arguments that were put forward in the case of *Cooper* v. *Swift*. The analysis showed that there was woven material in the sample, and by putting in even 8 per cent. of woven material the appellants had brought it within the definition of the Act.

After consultation, Lord Parmoor, the Chairman, said that they regarded the case as one of great importance. They accepted the evidence that the Hessian cloth was only used as a carrier to take the material through the machine. Although the proportion of woven material was found to be very small, they felt that it did come within the Act; therefore the conviction of the Court below must be upheld.

Mr. Robertson thereupon applied to the Court to state a case for the High Court, and Lord Parmoor said that they would agree to do this.

Government of Madras.

REPORT OF THE CHEMICAL EXAMINER FOR THE YEAR 1923.*

THE report of Dr. Clive Newcomb on the work of the Chemical Examiner's department shows that 3973 analyses and examinations were made, as against 3824 in 1922. The number of general analyses increased from 570 to 688, mainly owing to work for the Customs department, and a fresh record was established for the number of stain cases (1655 as against 1583 in 1922).

HUMAN POISONING CASES.—In the 204 cases of suspected human poisoning investigated, poison was detected in 92, giving a percentage of detections of 45·1 per cent., as compared with 49·1 in 1922.

The poison most commonly found was again mercury, with 27 cases, and arsenic next, with 20 cases. Opium was found in 12 cases, aconite in 11, oleander in 6, strychnine in 5, and atropine in 4. There was one case each of powdered glass, nitric acid, lysol, and kerosene oil.

Datura, which was found in 4 cases, is not a very common poison in Madras, and in most instances it is used as an aid to robbery. In one of the cases the drug was given in coffee, and the victim, feeling giddy some 20 minutes later, suspected foul play and reported the matter to the station police, who arrested the two accomplices. On them were found three packets of yellow seeds, and from each of these a poisonous mydriatic alkaloid was extracted. The victim recovered after 5 days in hospital.

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* G.O. No. 693, P.H., May 3, 1924.

In another case, a woman took datura for "stomach ache," and died 12 hours later, in spite of medical treatment. A mydriatic alkaloid was found in the stomach and contents, but not in the urine, which was collected *post-mortem.*

In the case of aconite poisoning a man was given a sweetmeat made of sugar and coconut and shared this with an acquaintance. Both were taken very ill with typical symptoms of aconite poisoning, but recovered under treatment. The vomits of both victims yielded, on extraction, a substance giving the reactions of an alkaloid, fatal to a frog, and, when applied to the tongue, producing tingling, followed by numbness. This was reported as a poisonous alkaloid giving the physiological reactions of aconitine. In this case the question arose whether it was likely that anyone would go on eating a substance the poisonous nature of which was indicated by the tingling of his tongue. When one tastes aconite, however, the tingling does not come on for some ten minutes, and full numbness often does not develop for half an hour or longer.

In another a man died after eating a curry. The curry contained an alkaloid giving the physiological reactions of aconite, and the symptoms of the victim were those of aconite poisoning. No aconitine could be detected, however, in the stomach or intestines, and it is suggested that the case should be put on record as one where aconitine was not found.

CATTLE POISONING.—Forty cases were investigated, and poison was detected in 17. The active principle of yellow oleander (thevetin) was the poison most commonly found (8 cases), and arsenic was second (7 cases). Mercury was found in 2 cases.

STAIN CASES.—During the year 380 cases were investigated for blood or semen or both. Of these, 353 (with 1549 articles) were examined for blood alone, and it was detected in 306 cases and 1050 articles. Of the 1018 specimens of blood stains submitted to the Imperial Serologist, at Calcutta, 924 were reported to be human blood, 8 ruminant animal blood, 2 bird's blood, 5 non-mammalian blood, and 7 blood non-human. In 65 cases the origin of the blood could not be determined, and in 17 cases the amount of stain was insufficient for the tests.

In one of the cases two pieces of white-washed wall were submitted on which were, what at first sight, appeared to be two prints of blood-stained hands left behind by the murderer. The prints on examination, however, did not give any of the reactions for blood.

This raised the question of the possibility of blood stains on white-washed walls becoming altered in the course of a few weeks, so that they would no longer answer to the reactions for blood. Stains were, therefore, made on a white-washed wall protected from direct sunlight, and exposed for a year at 25° to 35° C.

The appearance of the stains did not alter after the first 48 hours, and up to two months no difficulty was experienced in getting any of the reactions for blood. After a year the stains still gave a blue coloration with guaiacum and turpentine, and when treated with 10 per cent. potassium cyanide solution and ammonium sulphide and examined under the microscope, still gave the spectrum of cyano-hæmoglobin, though the colour was not the bright rose-pink of fresh stains, but of a browner shade. The reduced hæmatin spectrum could not be obtained after this interval, even when the stain was soaked for some time in water before boiling with potassium hydroxide and reducing with ammonium sulphide. As the other test is conclusive, it seems a safe inference that a blood stain on white-wash, if kept dry, will be detectable up to one year, at least.

SEMINAL STAINS.—In all the seminal stain cases Florence's test in the form described in last year's report (ANALYST, 1923, **48**, 490) was used. Certain stains, originally proved to be seminal, were kept for over a year in an envelope, and, although no spermatozoa could be found in them after they were a day or so old, they still gave a pronounced reaction in Florence's test after the whole period.

Florence's reagent has been found to be liable to lose some of its iodine if kept long, and should be made up fresh, or re-saturated with iodine, every few months. The reagent was also found useful in showing up the nature of paste stains, which may resemble seminal stains.

Ministry of Health.

INTERIM REPORT OF THE FOOD PRESERVATIVES COMMITTEE ON THE TREATMENT OF CHILLED BEEF AND OTHER FOODS WITH FORMALDEHYDE.*

THE Departmental Committee on the Use of Preservatives and Colouring Matters, appointed in July, 1923, was requested to make a special report on a process (named after its inventor the Linley Process) for the preservation of chilled beef by means of formaldehyde, but decided to issue an interim report dealing with the use of formaldehyde for food in general, and to devote special attention to its particular application as a preservative for chilled beef.

It is pointed out in the report, which is dated June 12th, that formaldehyde, when ingested, exerts an irritant action upon mucous membranes, and, after prolonged use, appears to cause inflammatory changes in the liver and also in the kidneys, where a portion of it is excreted. In the body it is partly oxidised to formic acid and partly excreted unchanged; in its passage through the body it combines with tissue proteins, and this combination renders its excretion slow, so that it is probably cumulative, and this feature makes its use as a preservative of food particularly objectionable. Formaldehyde may retard digestion, even when present in food in comparatively great dilution, and it combines with the protein constituents of foods, forming a compound which is less digestible than the original substance. Even if this formaldehyde-protein compound is ultimately digested and the products of digestion are absorbed, it is possible that these products are abnormal in character, and present knowledge does not justify us in asserting that they could be regarded as unobjectionable and without effect upon the normal metabolic processes of the body. On the contrary, recent advances in our knowledge of biological processes point to the far-reaching importance of the effects produced upon metabolism and nutrition by extremely minute amounts of substances ingested and absorbed, and, in view of this, a substance such as formaldehyde, which is known to produce gross changes in the composition of food materials and to be inimical to life, even in high dilution, must be regarded with grave suspicion in regard to its possible ultimate effects upon the normal processes of metabolism.

Formaldehyde may apparently be used for other purposes than those of merely preserving fresh food. Dr. (now Sir) G. S. Buchanan in a report to the Local Government Board, 1909, refers to its possible use for concealing incipient decomposition and for hiding any smell of staleness or putrefaction. Quite apart from the question whether formaldehyde is undesirable as a preservative for fresh food, its use for the purpose of foisting upon the consumer an article which has already begun to decompose is unquestionably reprehensible.

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* H.M. Stationery Office. 1924. Price 2d. net.

From the evidence given before the Committee, it would appear that the undesirability of using this substance as a preservative is generally recognised by traders. When formaldehyde is added to food nowadays, it is usually done by irresponsible persons, and its use as a food preservative appears to be comparatively rare and sporadic. Although butchers are being supplied, to some extent, with a solution of formaldehyde for treating the surface of meat, it is possible that they may be unaware of the material they are using. It may be added that, with scarcely an exception, every country which has made legal enactments or regulations in connection with the purity of its food supply has prohibited the addition of formaldehyde to food, either specifically, or implicitly in a general prohibition of preservatives.

Hence, having regard to the interests of the trader and of the consumer, the Committee have no hesitation in recommending that the addition of formaldehyde or any of its derivatives to food or drink should be absolutely and specifically prohibited.

The Linley Process.—The object of the Linley process is to enable beef to be carried chilled at a temperature of about 29° to 30° F. from distant countries, such as the Argentine and Australia, so as to arrive in this country in good condition and free from moulds which are apt to develop on the surface of lightly chilled beef after the lapse of a certain time. The process was in operation some years before the war in connection with the transport of chilled beef to this country from the Argentine, but by no means all of the Argentine chilled beef sent here was treated by the process. The Linley apparatus was installed in several abattoirs in the Argentine, but none of the abattoirs in that country controlled by United States firms ever adopted the process, and without its aid these firms were able to export chilled beef to England successfully. During 1909 and 1910 five trial consignments of chilled beef treated by the Linley process were sent from Australia, but after that date shipments were discontinued.

Early in 1923 the Argentine Government had under consideration the question as to whether the Linley process should be recognised, but, before making any pronouncement on the matter, consulted the British Government, and were informed that the revival of the process would not be in the interest of the consumer. In a decree dated February 20, 1923, the Argentine Government prohibited the treatment of meat by the Linley process.

The chief reason given for desiring the resumption of the process is that it will enable the establishment of a trade in chilled beef from Australia, since, under present conditions, the journey from Australia is too long to permit the successful transport of chilled beef to this country without the aid of some such process. For the information of the Committee, and in order to demonstrate the feasibility of carrying chilled beef treated by the Linley process from Australia, a firm of importers interested in the process offered to arrange for a trial consignment to be sent to London. It proved impossible to make the necessary arrangements in Australia, and the consignment was shipped from New Zealand instead. On April 1 the consignment arrived at the Port of London; the hold was opened on April 3, and the beef was inspected by the Committee as it was removed from the hold. The quarters were frozen hard, and there were copious growths of mould upon them. The whole consignment was subsequently condemned by the Port Medical Officer as unsound and unfit for food, and was surrendered by the importer for destruction.

Samples were taken from the exposed surface of muscular tissue of both hind and forequarters and were examined by Dr. G. W. Monier-Williams, for the presence of formaldehyde. The method used was that elaborated by Dr. Schryver and described in the Local Government Board's Food Reports, No. 9, 1909. The amount of formaldehyde found was relatively small, but it is noteworthy that traces were detected as deep as 30 millimetres from the surface of the meat, showing that formaldehyde penetrates into the meat with ease and does not confine itself merely to the surface. Analyses made on behalf of the Local Government Board of chilled beef treated by the Linley Process and imported into this country before the process was discontinued also demonstrated the readiness with which formaldehyde penetrates into the meat in this process.

The inventor ascribed the failure of the experiment to several causes, the chief of which were that the cattle had not been properly slaughtered and that sufficient care was not taken in the subsequent dressing and handling of the carcasses, that the meat had not been properly hung, and that the fan for circulating the air in the hold had not been working effectively.

The Committee think it fair to state that the conclusions at which they have arrived have not been influenced by the failure of this experiment.

If it were justifiable to assume that formaldehyde was sufficiently unobjectionable to enable it to be tolerated in small quantities in foods, it might be possible to permit its use in connection with chilled beef and to prescribe a limit for the amount which might be present, say, 2 to 3 parts per million in a surface layer of beef 7 to 10 millimetres thick. There would, however, be considerable practical difficulties in the equitable enforcement of such a prescription.

Moreover, declaration would be a necessary corollary to treatment with formaldehyde, and meat traders in this country would have to be allowed to treat their beef in similar fashion, if they so desired.

It appears possible that a section of the trade might benefit financially by the treatment of chilled beef with formaldehyde, but we think that the true interests of the trade as a whole would not be furthered by a revival of the practice. There appears to be much in the statement that the treatment of beef with formaldehyde may correct, in part at any rate, improper and defective methods of preparation, handling, storage, and transport, but it is undesirable that preservatives generally should be used to support the perpetuation of bad methods in dealing with foodstuffs. The Committee are satisfied that the consumer will not be prejudiced by their non-interference with existing conditions in the meat trade, and that no ultimate advantage to the consumer would accrue from a revival of the Linley Process.

Having regard to all the information which has been given to them, they are of opinion that no exception in favour of the Linley Process should be made from their general condemnation of the use of formaldehyde in any article of food or drink.

Deaths.

We regret to have to record the deaths of two of our Members:

Sir George Beilby, August 1st.
Lionel Gowing-Scopes, August 16th.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Estimation of Moisture in Wheat and Flour. H. Snyder and B. Sullivan. (*Ind. Eng. Chem.*, 1924, **16**, 731, 744.)—In twenty-eight comparative estimations of moisture in flour, 1·43 per cent. more moisture was found by drying the samples for five hours at 105° C. than when they were dried for the same length of time in an ordinary water-oven. A second similar series of tests showed that drying at 100° C. under reduced pressure (600 to 750 mm. "vacuum") yielded 1·67 per cent. more moisture than was found by drying in the water-oven. W. P. S.

Estimation of Egg Solids in Alimentary Pastes (Noodles). R. Buchanan (*J. Assoc. Off. Agric. Chem.*, 1924, **7**, 407–424.)—Juckenack's method, consisting in estimation of the phosphoric acid in the hot alcoholic extract, was, until recently, regarded as the most reliable procedure for the estimation of eggs in egg products, but Rask, Hertwig (ANALYST, 1924, 37) and others find that this method results in incomplete extraction. More complete extraction is effected either by digestion, first with hot ammoniacal alcohol and afterwards with ether (Rask and Phelps), or by digestion with 70 per cent. alcohol, followed by extraction with a mixture of alcohol and ether (Hertwig). Hydrolysis by means of hot hydrochloric acid and extraction of the fat from the resulting solution has been suggested, but organic phosphoric acid is decomposed in this way.

Alimentary pastes have been prepared from semolina, flour, whole eggs or yolks, either dried or frozen, and dried albumin, mixed in various proportions, the resulting products being analysed for lipoids and lipoid P_2O_5 by the Rask and Phelps and the Hertwig methods, and for the moisture, total nitrogen, water, soluble nitrogen, and water-soluble protein nitrogen precipitable by 40 per cent. alcohol. Ratios between certain of these substances, mulplied by 100, as proposed by Hertwig, distinguish clearly between whole egg and yolk noodles, and also between these and flours and semolinas. The recovery in the whole egg and yolk noodles of the lipoid P_2O_5 of the ingredients ranged from 81·3 to 104·5 per cent. by the Rask and Phelps method, and from 80·1 to 105·5 per cent. by the Hertwig method. If use is made of the factor 1·1 to compensate for loss of lipoid P_2O_5 during manufacture of the noodles, a close approximation is attained to the actual whole egg and yolk solids content. The formulae and basic values employed in the calculations, which are those of Hertwig, all refer to dry matter and are as follows:—Average percentages of lipoid P_2O_5 in flours, whole eggs, and commercial yolks are respectively 0·055, 1·38, and 1·78; percentage of whole egg solids in any sample of noodles is thus given by $\frac{(A-0{\cdot}055)\,100}{1{\cdot}38-0{\cdot}055}=75{\cdot}5\,(A-0{\cdot}055)$, where A represents the percentage of lipoid P_2O_5 found, multiplied by 1·1. Similarly, for samples containing commercial yolk, the percentage of commercial yolk solids will be 58·0 (A – 0·055).

Noodles stored for about a year in either sealed jars or paper boxes showed considerable and similar losses in lipoid P_2O_5, but only slight change in the proportions of water-soluble protein nitrogen precipitable by 40 per cent. alcohol.

T. H. P.

Separation of Trimyristin from Milk. M. Piettre and C. Roeland. (*Compt. rend.*, 1924, **278**, 2283–2285.)—When milk fat is dissolved in a mixture of ether and alcohol and allowed to evaporate slowly, well-defined needle-shaped crystals, melting at 52–54° C., are obtained in 24–48 hours; these have been identified as trimyristin. The crystals are insoluble in water, slightly soluble in alcohol, and readily soluble in ether; they may be purified by recrystallisation from alcohol-ether, about 2 grms. of crystals being obtainable from 1 litre of milk. The best procedure is to mix the cream from 500 c.c. of fresh milk with 200 c.c. of Adams' mixture (alcohol 75 per cent., 1000 c.c., ether 1100 c.c.) and, after separation, remove the upper layer (which consists of an alcohol-ether solution of the fat), filter it, and allow it to evaporate at 8 to 10° C. H. E. C.

Estimation of Raffinose in Sugars. Proportion of Raffinose in Molasses. E. Saillard. (*Comptes rend.*, 1924, **178**, 2189–2192.)—In test experiments with 7 to 8 per cent. solutions of pure raffinose inversion was found to be (by Clerget's method) 51·56 at 20° C. Taking the inversion of sucrose as 34 at 20° C., S the percentage of sucrose, γ that of raffinose, A the direct polarisation, B polarisation after inversion and C the sum of A and B, $A = S + \gamma$, $B = -0{\cdot}34S + 0{\cdot}5156\gamma$; whence, $\text{sucrose} = C - 0{\cdot}4844A/0{\cdot}8556$, and $\text{hydrated raffinose} = A - S/1{\cdot}57$. In the case of molasses sucrose is usually calculated from the formula $S = (A + B) \times 100/144 \times 1/2t$. As a rule A is greater than S, and the nitrogen and raffinose content influence this difference, raffinose being probably formed to a greater extent at the end of the season, or in the beet silos. D. G. H.

Estimation of Reducing Sugars and Sucrose. W. Thomas and R. A. Dutcher. (*J. Amer. Chem. Soc.*, 1924, **46**, 1662–1669.)—The method of Benedict and Osterberg (*J. Biol. Chem.*, 1918, **34**, 195) for the estimation of sugars in urine has been adapted, with modifications, to the estimation of reducing sugars and sucrose in plant extracts. This colorimetric method is superior to the gravimetric, volumetric and optical methods for the estimation of small amounts. The following reagents are necessary:—(1) The picrate-picric solution.—To 500 c.c. of 1 per cent. sodium hydroxide solution are added 36 grms. of picric acid (dried at 60° C. and purified) and 400 c.c. of hot water. The mixture is shaken, cooled and diluted to 1 litre. (2) The mercuric nitrate solution.—A solution of 110 grms. of mercuric oxide is added gradually to 80 c.c. of concentrated nitric acid, stirred, heated to boiling, cooled, and to it 30 c.c. of 5 per cent. sodium hydroxide solution are added, and the whole made up to 1 litre. A representative sample (about 50 grms.) of the plant parts is plunged into 95 per cent. boiling alcohol free from acids and aldehydes, filtered, washed with 75 per cent. alcohol, dried in an oven at 70° C. and pulverised. The sugars are removed by extracting the powdered plant in a Soxhlet apparatus, using the alcoholic filtrate, until the extract is colourless. A 20 c.c. to 100 c.c. aliquot portion, containing from 0·025 to 0·150 grm. of sugars, is evaporated to remove the alcohol, and the residue dissolved in 100 c.c. of water. A slight excess, say 10 c.c., of mercuric nitrate solution is added; then small quantities of solid sodium bicarbonate are added gradually, with stirring, until all frothing ceases. The solution must be kept just alkaline. It is then filtered into a 250 c.c. flask, the precipitate washed with 5 per cent. sodium bicarbonate solution, and the solution and wash liquid made up to the mark and shaken. From 30 c.c. to 50 c.c. are treated with 0·3 to 0·5 grm. of zinc dust in a 75 c.c. test-tube, one drop of concentrated hydrochloric acid added, the tube stoppered and left for 15 minutes and the contents then filtered. From 5 c.c. to 10 c.c. of the filtrate, tested to ascertain the absence of mercury, are pipetted into a 50 c.c. pyrex test-tube, 10 c.c. of the picrate-picric solution added and 2 c.c. of a 25 per cent. sodium carbonate solution. Tubes containing this solution and a standard colour solution simultaneously prepared (10 c.c. of standard glucose solution, 10 c.c. of the picrate-picric solution and 2 c.c. of the sodium carbonate solution) are plugged and immersed for 20 minutes in a water-bath at 95°, removed, cooled to room temperature, and the liquids compared in any standard colorimeter. The products of hydrolysis of sucrose are equivalent amounts of glucose and fructose, and, since sucrose up to 0·1 *M* concentration has no picrate-picric colour, it is possible to estimate the quantity present in solution not exceeding this, by the increase in colour after inversion. Mercuric nitrate is better than lead acetate as a clarifying agent. P. H. P.

Estimation of Starch. W. Thomas. (*J. Amer. Chem. Soc.*, 1924, **46**, 1670–1675.)—A colorimetric method for the estimation of starch by means of conversion into glucose and maltose by the mixed enzyme taka-diastase—secreted by *Aspergillus oryzæ*—without a secondary hydrolysis is described. Under the conditions of conversion, the ratio of dextrose to maltose is remarkably constant, and the analytical error introduced by regarding this ratio as 2·0 is very small, since the picrate-picric reducing ratio of dextrose to maltose is relatively high. From 1 to 4 grms. of the vacuum-dried residue, freed from sugars as above, is gelatinised by heating to boiling for 40 minutes with 200 c.c. of water, with stirring, cooled to 38° C. and incubated at this temperature, with occasional stirring for 24 hours with 0·1 grm. of the enzyme preparation and 2 c.c. of toluene. Toluene lost through evaporation is replenished. The solution is then heated on a water-bath to boiling for 15 minutes to render the enzymes inactive, and the residue is filtered and thoroughly washed by decantation with water into a carefully calibrated 250 c.c. flask. An aliquot portion (30 c.c.) is taken, 5 c.c. of the mercuric nitrate reagent and sodium bicarbonate are added, followed by filtration and treatment with zinc and hydrochloric acid, as in the preceding abstract. One to 5 c.c. is diluted to 10 c.c. in a pyrex test-tube, reagents are added, and the solution is compared with a standard colour solution, as before. The dilution factor for mercuric nitrate solution is used, and a blank estimation on 0·1 grm. of taka-diastase is carried out simultaneously. The recovery by this method of 0·25 grm. of potato starch added to (1) spurs and (2) leaves of apple trees in 6 experiments was from 98·4 to 99·4 per cent. Experiments on a secondary hydrolysis with hydrochloric acid and a method for the estimation of other "reserve" polysaccharides are described. P. H. P.

Detection and Estimation of Small Quantities of Cyanogen in Wine. F. Mach and M. Fischler. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 329–337.) —Kolthoff's method (*Zeitsch. anal. Chem.*, 1918, **57**, 3) is the most sensitive for the detection of the cyanogen which may be present in wines after fining with ferrocyanide and iron. To 10 c.c. of the wine are added 20 mgrms. of ferrous sulphate and 10 drops of a solution containing 8 grms. each of sodium carbonate and bicarbonate in 100 c.c.; the mixture is allowed to stand for half an hour and then acidified with sulphuric acid; 2 mgrms. of cyanogen per litre are thus readily detected. For the estimation the aeration method, followed by iodimetric titration, is the best; 50 c.c. of the wine are placed in each of two wash bottles connected in series with two others, the first containing 20 c.c. of 4 per cent. bicarbonate solution saturated with carbon dioxide, and the second 20 c.c. of *N* sodium hydroxide solution. A stream of air is aspirated through the bottles for an hour, and then the sodium hydroxide solution is neutralised by the addition of 20 c.c. of *N* sulphuric acid, and any excess of acid is removed by the addition of a little calcium carbonate; finally the liquid is titrated with 0·01 *N* iodine solution. Working on small quantities of cyanogen the results are always a little low, about 80 per cent. of the true quantity being indicated. Small distributing arms may, with advantage, be fitted to the inlet tubes of the wash bottles. Solutions of tartaric or acetic acids, or wine, always form small amounts of hydrocyanic acid with ferrocyanide at summer temperatures, a trace of this acid is therefore always present after fining with this salt. H. E. C.

Hydrocyanic Acid in Distilled Wines. O. Reichard. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 339–349.)—The fining of wine by ferrocyanide, which was formerly forbidden, is now allowed in Germany; as the result of a number of experiments on distilled wines it is concluded that those fined by this substance almost invariably contain traces of hydrocyanic acid. Details are given of various experiments on the fining of different kinds of wine with ferrocyanide. The presence of traces of hydrogen cyanide cannot now be taken as evidence of the presence of spirit distilled from stone fruit; such addition can only be recognised by the identification of the characteristic aromatic compound or ester, as well as of hydrocyanic acid. The ester may sometimes be identified by shaking out with chloroform. H. E. C.

Hastening the Coloration of Lemons. F. E. Denny. (*J. Agric. Res.*, 1924, **27**, 757–769.)—Commercially mature lemons are frequently green when picked, and, in order to produce the usual yellow colour rapidly, they are placed in rooms heated by the fumes from kerosene stoves. Investigation showed that the effective colouring agent is an unsaturated hydrocarbon, probably ethylene, although this gas was not positively identified among the products of the combustion. Ethylene in concentration 1:200,000 colours the lemons at 50–60° F. in 5 to 8 days, or in 6 to 10 days at concentration 1:2,000,000; the presence of oxygen is essential. The rate of respiration and the output of carbon dioxide are much increased by the ethylene or stove gas. The absence of physiological or explosive properties in such high dilution render ethylene quite a satisfactory agent for the yellowing of lemons, and, although a large number of other substances were found to have the property in varying degree, they all either injure the fruit or have some deleterious effect. (*Cf.* ANALYST, 1924, 284.) H. E. C.

Estimation of Essential Oils in Certain Flavouring Extracts and of Camphor or Peppermint Oil in Certain Pharmaceutical Preparations. W. W. Randall. (*J. Assoc. Off. Agric. Chem.*, 1924, 7, 425–430.)—The method in which the essential oil or camphor is precipitated from its alcoholic solution by means of acidified calcium chloride solution and then shaken with petroleum spirit boiling at 40° to 60° C. is attended by various difficulties and inaccuracies, which the author now shows how to avoid. Use is made of: (1) A solution of density about 1·30, prepared by dissolving calcium chloride in water, mixed with about 4 per cent. of its volume of concentrated hydrochloric acid, and filtering perfectly bright the next day; a large bottle provided with a siphon, at the end of which is a tap with a drawn-out tip, forms a convenient container. (2) Redistilled light petroleum spirit boiling at 40° to 60° C. (3) Calibrated 8 per cent. Babcock milk-test bottles. (4) Pipettes of 10, 5, 1 and 0·5 c.c. capacity, the last two being of thick-walled tubing of 2 to 3 mm. bore, drawn out to a long tip, which is bent slightly and filed flat at the end so as to deliver the petroleum spirit smoothly against the inner wall of the neck of the milk-test bottle.

With extracts containing about 5 per cent. of oil, such as orange or lemon extract, or about 3 per cent. of oil, such as peppermint or anise extract, 10 c.c. are pipetted into a clean Babcock bottle, which is then filled to the shoulder with the calcium chloride solution and shaken; exactly 1 c.c. of the gasoline is next run in, care being taken that the tip of the pipette does not touch a wet spot on the neck of the bottle. The latter is stoppered tightly with a soft cork previously wetted with the calcium chloride solution, and afterwards shaken violently for 1 or 2 minutes and centrifuged at fairly high speed for 2 minutes. The cork is then removed and enough calcium chloride solution quickly added to bring the whole petroleum spirit column within the graduated part of the neck, the bottle being then corked, inverted, shaken vigorously and centrifuged again for 3 to 5 minutes. The bottle is then placed upright and the volume of the petroleum spirit column read, the mean graduation between the upper and lower meniscus being taken. The lower meniscus should be flat and sharp, any whitish "collar" indicating lack of sufficient acid in the calcium chloride solution. A parallel estimation in which 10 c.c. of alcohol are used instead of the extract serves to measure the volume of gasoline delivered by the pipette in terms of the bottle graduation; deduction of this volume from that of the petroleum spirit and oil solution, and multiplication of the remainder by 2 gives the percentage of oil in the extract. For extracts containing 7 to 15 per cent. of camphor or essential oil, such as spirits of camphor or essence of peppermint, 5 c.c. instead of 10 c.c., and the multiplier 4, are used. For extracts containing as little as 2 per cent. of essential oil, *e.g.* nutmeg extract, not more than 0·5 c.c. of gasoline is used to 10 c.c. of the extract.

The factor used may vary somewhat with the composition of the gasoline, which should be tested by an estimation of the oil in a standard 5 per cent. orange extract.

T. H. P.

Detection of Aldehydes in Ethers for Anæsthesia. E. Isnard. (*J. Pharm. Chim.*, 1924, **29**, 5–9.)—The methods given by the various pharmacopoeias for the detection of aldehydes in ether differ widely in sensitiveness, and the following three tests are suggested. (1) For ordinary purposes.—Four c.c. of rosaniline bisulphite (Schiff's reagent) added to 10 c.c. of the ether, vigorously shaken 5 or 6 times, and left for 15 minutes, should not produce a rose-violet coloration (sensitiveness 1 in 10,000). (2) For the purest ether.—Ten c.c. of ether shaken with 2 c.c. of Nessler's reagent should not give, in 5 minutes, any red-brown precipitate turning black on adding 0·1 N potassium cyanide solution (sensitiveness 1 in 200,000). (3) For the purest ether.—Ten c.c. of ether shaken with 3 c.c. of ammoniacal silver nitrate solution containing sodium hydroxide should not give, in 5 minutes, any brown-black coloration (sensitiveness 1 in 75,000).

D. G. H.

Cascara Content of the Wood and Bark of *Rhamnus Purshiana*. R. H. Clark and K. B. Gillie. (*Amer. J. Pharm.*, 1924, **96**, 400–412.)—Physiological tests showed the average potency of extracts from the wood to be 38 per cent. and from the bark 53 per cent., and it is concluded that the wood contains sufficient of the active constituents to make it a practicable source of commercial extracts. The active principle, since complete hydrolysis does not destroy its activity, is probably not a glucoside, although possibly the hydrolytic product of one. Average values obtained for the bark (B) and wood (W) extracts were found to be:—Sp. gr., (B) 1·072 (W), 0·985; total solids, (B) 27·4, (W) 4·69 per cent.; ash, (B) 1·08, (W) 0·27 per cent.; and reducing sugars, before hydrolysis, (B) 5·15, (W) 0·947 per cent.; after hydrolysis, (B) 6·88, (W) 1·5 per cent.

D. G. H.

New Reaction for Myrrh. S. Dezani. (*Giorn. farm. Chim.*, 1924, **731**, 5; *Chem. Abstr.*, 1924, **18**, 1177.)—Myrrh gives an intense violet to azure blue colour, persisting for several hours, when treated with a reagent consisting of chloroform (8 parts), glacial acetic acid, (3 parts), and ethyl acetate (1 part), followed by one or two drops of strong sulphuric acid. The reaction is sensitive to 0·5 mgrm. of myrrh. Nineteen other gums and resins examined gave either no coloration or else fugitive yellow, green or red shades.

Identification and Classification by Chemical Methods of Drugs containing Tannin. I. Application of the Goldbeater's Skin Test. C. J. Jordan and A. H. Ware. (*Pharm. J.*, 1924, **113**, 102–104.)—The goldbeater's skin test (*Biochem. J.*, 1922, **16**, 4; ANALYST, 1924, **49**, 25) is regarded as the best single general test for tannins so far devised. If the drug extractive stains the skin at all, the stain will be obtained before immersion in the iron solution, and is practically identical with that obtained by the treatment with ferrous sulphate and 5 per cent. hydrochloric acid. Extractives of certain drugs may give stains which mask, to a certain extent, the tannin reaction, but it is usually possible to distinguish the dyeing and tannin effects, and, at the same time, obtain valuable indications of the presence of natural vegetable dyes. "Dye" stains are not usually greatly enhanced by the iron treatment. Tables are given of (1) drugs giving positive results characteristic of tannin; (2) drugs giving poor or dubious results; and (3) those definitely showing absence of tannin. All the B.P. Codex tannin drugs fall within Class (1) except pyrethrum, which is in Class (2). D. G. H.

New Classificatory Tests for Drugs containing Tannins. A. H. Ware. (*Pharm. J.*, 1924, **113**, 104–105.)—(*a*) *Re-investigation of Stiasny's Test for Phlobotannins.*—The test, carried out as follows, was applied to all the substances classified for the goldbeater's test (see preceding abstract) with confirmatory results. A few drops of 40 per cent. formaldehyde solution are added to a few c.c. of extractive, and subsequently the same number of drops of 10 per cent. dilute hydrochloric acid, and the whole boiled for 1 minute (excess of formaldehyde and acid should be ensured). After cooling, and filtering, the precipitate is treated on the paper with successive portions of water, 90 per cent. alcohol, and aqueous alkali, when a copious residue indicates phlobotannin. To the filtrate are added 1 or 2 drops of 10 per cent. ferrous sulphate solution, and then, drop by drop, a 5 per cent. potassium hydroxide solution (unless the content of chromogenic phenol is very small or none, no precipitate of ferroso-ferric hydroxide occurs). Iron-greening filtrates are yielded by many anthoxanthine bodies; iron-browning filtrates by certain substances containing anthroquinone derivatives; and iron-blueing filtrates by substances containing gallic acid, gallotannins and certain anthocyanins.

(*b*) *New classificatory tests for Tannins with Iodine and Ammonia.*—On boiling the tannin-containing drug extractive with a slight excess of alcoholic iodine solution the following reactions may occur; (1) Phlobotannin drugs yield copious coloured precipitates, the filtrate, in the absence of phenolic bodies, becoming practically colourless on addition of ammonium hydroxide solution, and the precipitates, on treatment with ammonium hydroxide solution, being either soluble, with formation of coloured solutions, (*e.g.* iron-colouring bodies not precipitated by Stiasny's reagent, and non-iron colouring bodies); insoluble (separated into iron colouring and non-colouring); or partly soluble and insoluble. (2) Phlobotannin bodies containing gallic acid or gallotannin, or both, also give precipitates, but the filtrates are highly coloured on addition of ammonium hydroxide solution. (3)

Bodies containing only small quantities of tannin, characterised by reactions due to substances other than tannins. D. G. H.

Assay of Valerian Root and certain other Aromatic Drugs. K. Bullock. (*Pharm. J.*, 1924, **113**, 109–113.)—Fifty grms. of the drug (No. 40 powder) are mixed in a mortar with 50 c.c. of water and 80 grms. of dry sand, extracted for 4 hours with petroleum spirit boiling below 40° C., the solution dried over anhydrous sodium sulphate and filtered, and the bulk of solvent distilled. The residue (3 to 4 c.c.) is dried to constant weight (A) in a current of dry air, dissolved in 15 to 20 c.c. of 70 per cent. alcohol, the fat filtered off, the filtrate and washings transferred to a separating funnel, 20 c.c. of chloroform added, and the whole shaken. Excess of water containing sodium sulphate is added, the whole shaken, the chloroform layer separated, and the diluted alcohol shaken out with 2 successive portions of 5 c.c. of chloroform. The solution is dried, filtered, the solvent removed as before, and the residue weighed (B). Then the percentage of fat extracted is 2(A–B), and that of oleo-resin present in the drug 2B. A complete separation of the oil and resin in the unchanged condition is difficult to bring about, chiefly on account of resinification, but the best method was found to be to dissolve the oleo-resin in chloroform (which prevents bumping and local heating at the beginning) to add excess of glycerin (to prevent oxidation and to bring over last traces of oil; also it contains no water to cause hydrolysis of borneol esters), and to distil *in vacuo*. It is sufficient to distil 30 to 40 c.c. of glycerin for 1 grm. of oleo-resin. The quantity of water present in the drug at the time of extraction has an important effect on the yield of oil, and the fresh root must be dried or completely exhausted with alcohol of at least 98 per cent. strength, and the extract treated with petroleum spirit, preferably with the addition of concentrated hydrochloric acid. D. G. H.

Biochemical, Bacteriological, etc.

The Relation of P_H Value to Tungstic Acid Precipitation of Protein. A. T. Merrill. (*J. Biol. Chem.*, 1924, **60**, 257–259.)—Diphtheria antitoxin serum, peptone free, has been examined, and the protein-free nitrogen estimated by the tungstic acid method of Folin and Wu (*J. Biol. Chem.*, 1919, **38**, 81). At a P_H of about 5·0, which is on the acid side of the isoelectric point of serum globulin, the zone of maximum precipitation of nitrogen occurs. A value of 0·10 per cent. protein-free nitrogen is obtained in the filtrate. An estimation by the trichloroacetic acid method of Greenwald (*J. Biol. Chem.*, 1915, **21**, 61) gave identical results with those obtained by tungstic acid at the P_H of maximum precipitation. In peptone solutions the nitrogen precipitated by tungstic acid, increased considerably as the P_H decreased from 4·0 to 1·0, with a very slight reversal of precipitation at an acidity of 10 per cent. acid solution. The peptone nitrogen is only slightly precipitated at a P_H of 4·0, whilst at this acidity the protein in the serum is completely precipitated. There is no known P_H at which peptide nitrogen is precipitated separately from other nitrogen compounds in peptone solution. A change in the proportion of tungstic acid to peptone, or the concentration of reagents in solution, affects the quantity of nitrogen precipitated. It is suggested that the tungstic acid precipitate in peptone solutions is affected by the various peptones, bases, amino acids and other organic acids probably present in the peptone. Tables show the results obtained. P. H. P.

Estimation of Iodine in Food, Beverages and Excreta. J. F. McClendon. (*J. Biol. Chem.*, 1924, **60**, 289–299.)—Other methods for estimating minute quantities of iodine are discussed and some points on the difficulties of ashing are considered. Two convenient ways of evaporating about 100 litres of water, keeping it alkaline the whole time, are given. The dry sample is ignited in a boat in a silica combustion tube having a water-cooled stopper at one end, whilst the other end passes into a sodium hydroxide solution. This solution, and the rinsings of the tube, are evaporated to dryness, and the residue powdered, and mixed in a mortar with the powdered ash. The iodides and iodates are extracted by grinding the powder in a little water. This is filtered, and an accurately measured aliquot portion (say 7·5 c.c.) is taken, neutralised with concentrated hydrochloric acid, and the volume made up to 10 c.c. This is then transferred to a separatory funnel (12 c.c.), and shaken with 1 drop of concentrated hydrochloric acid and 1 c.c. of purified carbon tetrachloride. A pink colour denotes the presence of iodate, as well as iodide, in the ash. One drop of 0·1 *N* arsenious acid added and left for 20 minutes reduces any iodate. One drop of nitrosyl sulphuric acid is added to oxidise iodide to iodine and the funnel shaken vigorously for two minutes to extract the iodine. The carbon tetrachloride is run into a glass-stoppered bottle, and centrifuged to remove water droplets. It is then put into one cup of a Bausch and Lomb micro-colorimeter, and into the other cup is put carbon tetrachloride, 1 c.c. of which contains 0·1 mgrm. of pure iodine. The iodine extracted is estimated colorimetrically, and 1 c.c. of carbon tetrachloride again added to the funnel and another extraction made. By repeated extractions practically all the iodine may be recovered, and thus the total amount estimated. Details are given for purifying the carbon tetrachloride and the iodine for the standard solution. A method for ashing foodstuff is described for the estimation of iodine in the presence of large quantities of organic matter. It is possible to detect and roughly to estimate 0·001 mgrm. of iodine in 1 c.c. of carbon tetrachloride, but 0·01 mgrm., or more, is desirable in the sample for analysis. P. H. P.

Polarimetric Method for the Estimation of Diastatic Power. H. C. Gore. (*J. Assoc. Off. Agric. Chem.*, 1924, **7**, 364–367.)—A solution of Lintner's soluble starch containing 2 grms. of the air-dry starch per 100 c.c., and an infusion of the diastatic product to be tested of such concentration that 1 c.c. corresponds with 50 mgrms. of the sample, are prepared. The initial polarisation of the mixture of starch and diastase solutions is determined by mixing 50 c.c. of the starch solution with 0·5 c.c. of concentrated ammonia solution and 0·5 c.c. of the diastase solution in the order named and reading in the polarimeter at 21° C. A mixture of 100 c.c. of the starch solution with 1 c.c. of the diastase solution is left at 21° C. for a measured interval of time, so chosen that the fall in the polarisation is not greater than 3° Ventzke. A sample of 50 c.c. is then withdrawn, mixed with 0·5 c.c. of strong ammonia solution, left for 25 minutes, and then polarised in a 400 mm. tube at 20 to 21° C. If *d* represents the fall in polarisation, *t* the time in hours during which the action of the diastase on the starch proceeds, the diastatic activity on the Lintner scale is expressed by the formula, $L = \frac{100d}{2 \cdot 18t}$, 2·18 being the fall in polarisation observed in a 400 mm. tube produced by 1 c.c. of a diastase solution representing 50 mgrms. of a malt of diastatic power 100 Lintner acting on 100 c.c. of a starch solution containing 2 grms. in 100 c.c. for 1 hour at 21° C. Should the fall in polarisation observed be very small, the remaining solution may be digested for a suitable longer period calculated from the fall in polarisation.

If the results are to be expressed on Lintner's diastase scale instead of the Lintner malt scale, the 1 c.c. of the diastase solution should be so prepared as to represent 1·2 mgrms. of the diastase preparation to be tested. T. H. P.

Behaviour of Insulin towards Fehling's Solution. Knops-Niederhoff. (*J. Pharm. Belg.*, 1924, **6**, 160; *Pharm. J.*, 1924, **113**, 45.)—Insulin can be adjusted to the strength of a given standard by determining the equivalent of 1 unit to dextrose. The insulin is added to 100 c.c. of 1 per cent. dextrose at 38° C., kept at that temperature for 2 hours and boiled, and the proportion of dextrose left estimated by means of Fehling's solution. It was found that, in some cases, urines of patients undergoing insulin treatment contained something which hindered reduction, possibly insulin, and it was necessary to boil with Fehling's solution for 30 minutes to obtain a positive result. In such cases Pavy's solution was used for the estimation after boiling. Such urines may also fail to react to the direct test for acetone, and should be distilled, the ketonic bodies being tested for in the distillate. D. G. H.

Biochemical Detection of Galactose in mixtures of Galactose and Arabinose. M. Bridel and I. Charpentier. (*J. Pharm. Chim.*, 1924, **30**, 33–42.)—The presence of galactose in a reducing mixture containing galactose and arabinose can be shown by first measuring the fall in reducing power and rotation effected by adding emulsin to the alcoholic solution of the sugars, and then removing the arabinose by adding hydrocyanic acid in the presence of a trace of ammonia, whereby the ammonia salt of the *l*-gluconic acid is formed. The ethylgalactoside may then be crystallised out. D. G. H.

Association of Manganese with Vitamins. J. S. McHargue. (*J. Agric. Res.*, **1924**, **27**, **417–424**.)—The author shows a distinct parallelism between the distribution of vitamin and of manganese in plant and animal tissues. In the plant the manganese is essential for the synthesis of chlorophyll, and a relationship is indicated between its presence and the formation of vitamins. The effect of the polishing of rice, which is well known to remove the vitamin, is shown by the following results:—Manganese in rice bran, 350; in rice polishings, 100; in unpolished rice, 25; and in polished rice, 10 parts per million. Similarly, wheat bran contains 175 parts, whole wheat 40 parts, and patent flour only 10 parts. The livers of the hog, sheep, and cow contain larger proportions of manganese than do their other organs, but experiments on cod livers showed only traces of manganese. H. E. C.

Preservation of Stock Cultures of Micro-organisms. A. C. Thaysen. (*J. Inst. Brew.*, 1924, **30**, 349.)—The two methods given are designed to maintain, for a long time, strains of micro-organisms, unimpaired in any way, without continual transfer to fresh media. For non-sporing types the following method was found satisfactory:—Sterile media (broth, wort, or whey) are inoculated with a vigorous culture of the organisms to be tested and incubated for 24 to 48 hours, till well established. Then 0·2 grm. of sterile precipitated calcium carbonate is added to each, and the tubes, after being carefully shaken, are sealed and stored at room temperature. The various types tested were:—*B. coli communis, B. paratyphi, B. fluorescens liquefaciens, B. phosphorescens* Bernh., *B. volutans, B. Kützingianum, Streptococcus acidi lactici, Streptococcus hæmolyticus, Staphylococcus pyogenes aureus*, a *Saccharomyces* from brewers' yeast, and a red torula. After one year all survived unimpaired in every characteristic, except *Streptococcus hæmolyticus*, which died in less than fourteen days. All the other types retained their bio-chemical and morphological properties to the fullest extent. A parallel experiment, using calcium diphosphate in place of calcium carbonate, showed that the former was not so satisfactory In testing for survival it was found necessary to keep the sub-cultures under observation for about 8 days before finally deciding whether the organism had survived or not.

For sporing organisms carefully cleaned sea-sand is used. Four or five grm. lots are placed in test-tubes and sterilised. Two or three drops of a pasteurised culture, containing a large number of spores, are dropped on to the sand and distributed evenly throughout it. The tubes are placed in the incubator till dry, when they are sealed and stored at room temperature. The types tested were:—*B. mesentericus fuscus, B. subtilis, B. amylobacter, B. aceto-ethylicus, Penicillium glaucum, Aspergillus glaucus*, and *Rhizopus japonicus*. All were kept for one year, with the result that the sub-cultures made from the sand cultures were found to be distinctly superior to their parent cultures in their enzymic reactions.

R. F. I.

Relation of Bacteria to Growth-promoting Substances. S. R. Damon. (*Amer. J. Hygiene*, 1924, **4**, 408–409.)—Representative organisms of the various bacterial groups—the non-spore-forming bacilli, the spore-forming bacilli, the mucoid bacteria, and the acid-fast bacteria—were grown on a vitamin-free medium, collected and desiccated, and added to the ration of rats on a diet devoid of the water-soluble vitamin. The dried organisms were added in the proportion of 2·5 and 7·5 per cent. of the ration, and the results, as shown by the weight curves of the animals, indicated an absence of any growth-promoting substance, except in the case of the acid-free bacteria. These organisms, of which *B. timothy*, *B. smegmatis*, and the Mest Bacillus, were used, seem to contain definite growth-stimulating substances, of the type of the water-soluble *B* vitamin, as the weight curves of the animals at once closely approximated those of rats on a diet containing an adequate amount of that substance. Hence, it now seems reasonable to conclude that certain bacteria, at least, produce this vitamin in their growth processes.

Toxicological and Forensic.

Toxicity of Salts of Copper. J. Effront. (*Comptes Rend.*, 1924, **178**, 2152–2155.)—The toxicity of copper salts depends upon the power of absorption of copper by the particular foods present, and the reversibility of the absorption. One hundred grms. of pulp of potato absorbed 4·29 per cent. of anhydrous copper sulphate; leeks, 4·97; beetroot, 7·06; and turnips, 12·36 per cent., when a 7·215 per cent. solution of copper sulphate was used and the concentration of the pulp was 4 per cent. By increasing the dilution of the solution absorption is rendered more complete, and, under certain conditions, the action becomes reversible. For example, in the presence of acid, salts of calcium, and peptones the copper salt is liberated to different extents. Tryptic peptones (1 per cent.) may cause the liberation of as much as 40 per cent. of the copper absorbed. D. G. H.

Organic Analysis.

New Method of Elementary Analysis based on the Measurement of Gaseous Volumes. L. Hackspill and G. D'Huart. (*Bull. Soc. Chim.*, 1924, **35**, 800–803.)—The high results obtained by this method for the percentage of nitrogen (ANALYST, 1923, **48**, 504) are due to air adsorbed by the cupric oxide, and may, therefore, be rectified by re-oxidising the reduced copper, not in air, but in oxygen, and allowing it to cool in this gas; this oxidation is effected in the combustion tube itself. After the second evacuation (for the estimation of the hydrogen) oxygen from a cylinder is introduced by way of a lateral tube furnished with a cock, the temperature being maintained at 400° to 500° C. for some minutes; it is advisable to keep the oxide in oxygen after this treatment.

In order to reduce, as far as possible, the length of time necessary for the collection of the gases, the total volume of the apparatus has been diminished, and the Sprengel pump replaced by a mercury pump. The bore of the silica combustion tube has been reduced to 5 mm., and the substance is mixed, if necessary, with lead chromate. With these modifications the method gives good results, and the whole process occupies rather more than two hours if an accuracy of 1 per cent. suffices for the hydrogen estimation, or three hours if exact figures are required. T. H. P.

Quantitative Separation of Solid and Liquid Fatty Acids. D. Holde, M. Selim and W. Bleyberg. (*Chem. Zeit.*, 1924, **48**, 448.)—A method devised by Meigen and Neuberger (*Chem. Umschau.*, 1922, **29**, 337), depending on the solubility of the thallium salts of liquid fatty acids and the insolubility of the thallium salts of solid unsaturated fatty acids in aqueous alcohol in the presence of strong excess potassium hydroxide has been found to be, on the whole, impracticable in the form suggested. By precipitating the insoluble thallium salts with a 50 per cent. alcoholic solution of the potassium salts, however, it is possible to separate the solid saturated acids (stearic, palmitic and a mixture of both) from the liquid unsaturated acids (primarily oleic acid) quantitatively and in a pure state. The method is not suitable for the separation of solid unsaturated acids (*e.g.* erucic acid), since the thallium salts of these acids behave similarly to those of the solid saturated acids. It might, however, be used for the separation of arachidic and lignoceric acids, on the one hand, and linolic and linolenic acids, on the other hand. P. H. P.

Acetyl Value of Fats. E. Raymond and G. Glot. (*Comptes rend.*, 1924, **178**, 2098–2101.)—Zerewitinoff's method for determining the hydroxyl number of a fat by measuring the volume of the hydrocarbon formed by the action of an aliphatic organo-magnesium compound may be improved by using anisole, which is quite inert towards the Grignard reagent, as solvent for the reacting substances. If the number of mgrms. of potassium hydroxide required to neutralise the free acids of 1 grm. of the fat is denoted by n, and a is the hydroxyl value, the number of hydroxyls, x, contained in 1 grm. is given by $x = a - \frac{n}{56000} = a - 0{\cdot}0000179n$, and the acetyl value by $A = \frac{56000x}{1+42x}$. The index a is calculated by means of the expression $a = \frac{0{\cdot}000162\ \mathrm{VH}}{p\mathrm{T}}$, where V is the number of c.c. of methane, measured at the pressure H cm. and the absolute temperature T, liberated by p grms. of the substance.

To obtain exact results the fat must be freed from moisture by being kept in a desiccator over sulphuric acid or phosphoric anhydride for 1 or 2 days, and the magnesium methyl halide must be added gradually to the solution of the fat in dry anisole. The reaction vessel consists of a round 50 c.c. flask, surmounted by a very wide neck closed by a rubber stopper, through which pass a delivery tube connected with a Bunte burette, and a pipette fitted with a plug, so that the organo-magnesium compound may be allowed to fall, drop by drop, into the vessel. Correction of the gaseous volumes may be avoided by making all measurements at the ordinary temperature, but, if this is very low, the vessel may require gentle heating. Especially if recently prepared, the organo-magnesium compound rapidly absorbs atmospheric oxygen, but after a time this absorption greatly diminishes, so that the volume of the methane disengaged in a determination becomes constant and the use of an inert gas is rendered unnecessary. Slight absorption of gas by the organo-magnesium compound may occur, but a correction is easily applied. The time occupied by a determination never exceeds an hour, and the results obtained agree well with those given by the ordinary methods, which are far more tedious. T. H. P.

The Reaction of Iodine with Fats. B. M. Margosches and W. Hinner. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 349–355.)—It is shown that alcoholic or dilute acetic acid solutions of iodine, which are brown, react with the unsaturated acids of fats in the presence of iodic acid as do violet solutions of iodine. Aqueous solutions of iodine in potassium iodide also react quantitatively if some potassium iodate is added; with such solutions the results obtained for the iodine absorption are identical with those with Wijs or Hübl solutions, but the time taken is longer (24 hours are sufficient). The function of the iodic acid is to remove the hydriodic acid which would otherwise interfere with the absorption by the fatty acid. H. E. C.

Influence of Various Solvents on the Hanus Iodine Values of Cottonseed and Coconut Oils. H. J. Bankston and F. C. Vilbrandt. (*Ind. Eng. Chem.*, 1924, **16**, 707–708.)—Chloroform and carbon tetrachloride are the most satisfactory solvents for use in the determination of the iodine values of oils by the Hanus method; of the two, chloroform tends to give more uniform and slightly higher results in the case of cottonseed oil, and variation in the quantity of solvent is without appreciable effect. Ether, ethyl alcohol and benzene are useless as solvents in this method. With oils of low iodine value, such as coconut oil, the effect of variation in the solvent is less marked, but chloroform is the best solvent as regards uniformity in the results obtained. W. P. S.

The Phytosterols of Wheat Endosperm. R. J. Anderson and F. P. Nabenhauer. (*J. Amer. Chem. Soc.*, 1924, **46**, 1717–1721.)—The unsaponifiable matter from the fat extracted from wheat endosperm has been examined. Wheat endosperm contains at least two different sterols, namely, ordinary sitosterol, $C_{27}H_{45}OH$, and dihydrositosterol, $C_{27}H_{47}OH$, m.p. 144–145° C.; $[\alpha]_D^{20}$, $+25{\cdot}82°$. The dihydrositosterol from wheat bran appears to be identical with the saturated sterol that occurs in maize endosperm. The substance exists throughout the wheat endosperm, but the bran is particularly rich in this sterol. Full experimental details are given. A method for separating sitosterol from the saturated sterol by means of acetic anhydride and sulphuric acid with the acetyl derivative is described. The method of Bondzynski and Humnicki (*Z. physiol. Chem.*, 1896–1897, **22**, 396) for separating coprosterol and cholesterol, after brominating a mixture of the two, could not be satisfactorily adapted for the wheat bran sterols. P. H. P.

Estimation of Anhydrous Soap in Lubricating Grease. B. Joachim. (*Ind. Eng. Chem.*, 1924, **16**, 725–726.)—*Free Fatty Acid.*—Ten grms. of the sample are heated with 10 c.c. of alcohol and 90 c.c. of benzene, the hot solution is filtered, and the insoluble portion washed with hot benzene. Twenty-five c.c. of neutral

50 per cent. alcohol are added to the filtrate, and this is titrated with 0·1 *N* sodium hydroxide solution. *Fatty Acids from the Soap.*—Ten grms. of the grease are stirred and heated on a water-bath for twenty minutes with 3 grms. of finely-powdered potassium hydrogen sulphate, the mixture is cooled, extracted with benzene, the benzene extracts are filtered, the filtrate is diluted with alcohol, and titrated. The difference between this titration and the first is a measure of the fatty acids of the soap. *Soap.*—The neutralised solution of the fatty acids is diluted with water, the aqueous layer separated, evaporated to remove alcohol, and then acidified; the liberated fatty acids are extracted with ether and titrated with 0·1 *N* sodium hydroxide solution. This neutral solution is evaporated, and the residue of soap dried and weighed. If metallic soaps are present in the grease, the quantities of the metals must be estimated, and the corresponding quantities of metal soaps then calculated. W. P. S.

Relation between the Durability and Chemical Composition of Woods. L. F. Hawley, L. C. Fleck and C. A. Richards. (*Ind. Eng. Chem.*, 1924, **16**, 699–700.)—The relative resistance to decay of certain woods can be explained by the fact that they contain a larger quantity of substances which are toxic to fungi than do the less durable woods. The aqueous extracts of chestnut, oak, black locust, redwood, etc., are more toxic than are the extracts from maple, birch and alder; the hot water extracts are more toxic than the cold water extracts, and the extract from the heart wood is in each case more toxic than the extract from the corresponding sapwood. There appear to be no grounds for the assumption that the sapwood contains more starch, sugar, gums, etc., than the heart wood, or that these furnish food for fungi and further their growth. W. P. S.

Estimation of Benzene in Petroleum Spirit and Oil of Turpentine. J. Pritzker and R. Jungkunz. (*Chem. Zeit.*, 1924, **48**, 455–457.)—The authors base a method for the estimation of benzene in petroleum or turpentine on its reaction (previously described by Hofmann and Hochtlen, *Ber.*, 1903, **36**, 1149) with nickel ammonium cyanide. The reagent is prepared by mixing 5 grms. of nickel sulphate, dissolved in 20 c.c. of water, with 2·5 grms. of potassium cyanide in 10 c.c. of water, and adding 20 c.c. of a strong solution of ammonia; after the mixture has stood on ice for half-an-hour the crystals are filtered off, and dissolved as required, to form a saturated solution in 50 per cent. acetic acid. On adding this solution to the liquid under test, and shaking for a few minutes, a bluish white powdery precipitate is obtained in the presence of benzene. For quantitative purposes it is convenient to take 10 c.c. of the hydrocarbon mixture, which may contain 1 to 1·2 c.c. of benzene, and shake it for 5 minutes in a stoppered flask with 50 c.c. of the freshly-prepared reagent. The precipitate is filtered off with the aid of the pump, washed first with water, then once with alcohol and once with ether, dried, first by drawing air through it and then over sulphuric acid, and weighed. The precipitate, which has the formula $Ni(CN)_2NH_3C_6H_6$, contains 37·9 per cent. of benzene. H. E. C.

Behaviour of Sodium Sulphoricinate in the Official Method of Tannin Analysis. U. J. Thuan and L. Favre. (*J. Soc. Leather Trades Chem.*, 1924, 346.)—The authors draw attention to the fact that the Official Method is only an indication of the amount of material present in solution which is absorbed by hide powder under the arbitrary conditions laid down, irrespective of whether this material is a real tannin or not. For instance, sulphite-cellulose extract, though giving no precipitate with gelatin, is largely absorbed by hide powder, and thus counts as tannin. Sodium sulphoricinate acts in the same way. If mixed with formaldehyde, the more is absorbed by the hide powder the higher the amount of formaldehyde. The presence of quebracho lowers the quantity absorbed, but if mixed with chestnut extract an emulsion is formed, thus raising the figure for insoluble substances. Analytical results are given in twelve experiments on various mixtures of sulphoricinate etc. R. F. I.

Microchemistry of White Pigments and Inert Bodies as they occur Mixed in Paints. H. Green. (*Ind. Eng. Chem.*, 1924, **16**, 677–680.)—Methods for the necessary preparation of the sample for analysis are given, as well as microchemical tests for the individual pigments; a scheme for the qualitative analysis of a mixture of white pigments is also outlined. For the removal of oil, etc., from a paint, a drop of the sample is spread on a slide with the addition of a few drops of turpentine, the solvent is evaporated at 150° C., and the slide is then immersed in benzene. After a minute or two, the slide is removed and dried; the operation may be repeated, if required. The pigments adhere to the slide, and may be mounted in turpentine or dammar resin for microscopical examination. A portion of the pigment, after removal of the oil, etc., is reserved for microchemical tests. W. P. S.

Inorganic Analysis.

Estimation of Carbon Monoxide. P. Lebeau and Ch. Bedel. (*Comptes rend.*, 1924, **179**, 108.)—The usual reagent for absorbing carbon monoxide (cuprous chloride) has the disadvantage of requiring repeated contact because of the dissociation of the compound formed. The authors claim that this can be overcome by using a suspension of cuprous oxide in sulphuric acid containing β-naphthol. The reagent is prepared as follows:—Ten grms. of β-naphthol are placed in a 125 c.c. flask. To this is added a suspension of 5 grms. of cuprous oxide in a cooled mixture of 95 grms. of concentrated sulphuric acid and 5 grms. of water. The mixture is agitated for several hours and filtered through asbestos. The dark liquor obtained is left several days, decanted from the small deposit formed, and kept for use in well-stoppered flasks. This solution absorbs 18 times its volume of carbon monoxide, has no action on hydrogen, methane and its homologues, and can remain in contact with mercury for a considerable time without change. It is therefore suitable for use in the usual gas analysis apparatus. R. F. I.

Mixed Indicator for Carbonate-Bicarbonate Titrations. S. G. Simpson. (*Ind. Eng. Chem.*, 1924, **16**, 709.)—In titrating a mixture of alkali carbonate and bicarbonate the use of a mixed indicator consisting of thymol blue (thymol-sulphonphthalein) and cresol red (*o*-cresol-sulphonphthalein) is recommended as indicator for the first half of the titration; the colour of the thymol blue disappears, and is followed by a change from pink to orange-yellow, which shade is taken as the end-point, that is, the conversion of carbonate to bicarbonate. Bromophenol blue (tetra-bromphenol-sulphonphthalein) is then added, and the titration continued until the colour changes from blue to green—an indication that the solution is neutralised completely. The end-point in the case of the mixed indicator is much sharper than is that obtained with phenolphthalein. The indicator solutions should be prepared as follows:—Thymol blue, 0·03 grm. +1·42 c.c. of 0·05 *N* sodium hydroxide solution diluted to 25 c.c.; cresol red, 0·91 grm. +0·58 c.c. of 0·05 *N* sodium hydroxide solution diluted to 25 c.c.; bromophenol blue, 0·03 grm. +0·66 c.c. of 0·05 *N* sodium hydroxide solution diluted to 25 c.c. One volume of cresol red solution is mixed with 2 volumes of thymol blue solution, and 3 drops of the mixture are used for a titration; 4 drops of bromophenol blue solution are used for the second titration. W. P. S.

Mercuric Bromates in Analysis. G. F. Smith. (*J. Amer. Chem. Soc.*, 1924, **46**, 1577–1583.)—It has previously been shown by Smith (*J. Amer. Chem. Soc.*, 1923, **45**, 1115, 1417, 1666; ANALYST, 1923, **48**, 462) that the presence of the mercuric ion advantageously alters the usual course of oxidation reactions involving bromate by increasing the oxidation value. Mercuric bromates should be useful as oxidation agents. The preparation of normal and basic mercuric bromates by the interaction of mercuric perchlorate and sodium bromate in acid and practically neutral solutions is described. For the latter, mercuric perchlorate, prepared from mercuric nitrate and perchloric acid, is dissolved in a small volume of water and added slowly, with constant stirring, to a hot, half-saturated solution of sodium bromate. Proportions taken should be calculated from the following equation, with a slight excess of sodium bromate:

$$Hg(ClO_4)_2 + NaBrO_3 + H_2O \longrightarrow Hg(OH)BrO_3 + NaClO_4 + HClO_4.$$

Precipitation occurs upon the first addition of mercuric perchlorate. The solution and crystals are cooled to room temperature, decanted, and the crystals filtered and washed with water, with the aid of centrifugal drainage. The normal mercuric bromate is prepared by treating a hot 3 *N* perchloric acid solution, nearly saturated with sodium bromate, with an equivalent amount of mercuric perchlorate dissolved in 3 *N* perchloric acid; the crystals thus obtained, after cooling the reaction mixture to 0° C., are filtered off and washed with water, with the use of centrifugal drainage. With *N* perchloric acid solutions mixed normal mercuric bromate and hydroxymercuric bromate are prepared. The hydrolysis of normal mercuric bromate results in the formation of basic mercuric bromate and free bromic acid.

$$Hg(BrO_3)_2 + HOH \longrightarrow Hg(OH)BrO_3 + HBrO_3.$$

The relation of hydroxymercuric bromate to known mercuric salts with complex cations is pointed out; and the solubilities of the two bromates in various concentrations of nitric and perchloric acids at 25° C. have been determined. Basic mercuric bromate has been shown to be preferable as a reagent for volumetric bromate reactions because of its stability in 2 *N* nitric and perchloric acids.

P. H. P.

Estimation of Lead in the Presence of Tin and Antimony. E. Stelling. (*Ind. Eng. Chem.*, 1924, **16**, 748.)—Lead may be estimated in alloys containing tin and antimony, without previous removal of these two metals, if advantage is taken of the solubility of tin and antimony oxides in concentrated sulphurous acid and hydrochloric acid. One grm. of the borings is treated with 20 c.c. of concentrated nitric acid and 10 c.c. of water, and evaporated to dryness; the residue is then digested at 60° C. for five minutes with 50 c.c. of water saturated with sulphur dioxide. Twenty c.c. of concentrated hydrochloric acid are added, and the mixture is boiled; the tin and antimony oxides dissolve completely. After the addition of 20 c.c. of concentrated sulphuric acid, the mixture is heated until sulphuric acid fumes are evolved, cooled, diluted with 20 c.c. of water containing 10 per cent. of alcohol and 10 per cent. of sulphuric acid, and the lead sulphate then collected and washed with the same solution. W. P. S.

Volumetric Estimation of Fluorine. W. W. Scott. (*Ind. Eng. Chem.*, 1934, **16**, 703–706.)—The method proposed depends on the precipitation of the fluorine as calcium fluoride. In the case of minerals it is necessary to remove phosphates and sulphates by extraction with hot dilute acetic acid; the dried material is then fused for forty-five minutes with a mixture of sodium carbonate, potassium hydroxide and silica. After cooling, the fused mass is treated with hot water, the solution filtered, and the insoluble portion washed with hot water. The calcium in this insoluble portion may be estimated. The filtrate is heated to boiling and a slight excess of 0·25 *N* calcium acetate solution (12·51 grms. of calcium carbonate and 75 c.c. of glacial acetic acid per litre) are added; the mixture is then rendered slightly acid with acetic acid, heated for five minutes, diluted to a definite volume, and filtered. An aliquot portion of the filtrate is treated with a known volume of 0·25 *N* sodium oxalate solution, the precipitated calcium oxalate is collected, washed, and titrated in the usual way with potassium permanganate solution; the quantity of calcium required to precipitate the fluorine is thus obtained. Owing to the slight solubility of calcium fluoride, and possibly to the formation of a complex compound, the ratio of calcium to fluorine is 40:48, instead of the ratio represented by the formula CaF_2. W. P. S.

Detection and Estimation of small Quantities of Fluorine. R. J. Meyer. (*Chem. Zeit.*, 1924, **48**, 422.)—Lanthanum acetate in presence of ammonium acetate is an extremely sensitive reagent for fluorine; the precipitated lanthanum fluoride, however, always contains adsorbed lanthanum acetate. The precipitate may be dried without decomposition at 130° C. to constant weight. On subsequent ignition over a Bunsen burner the acetate in the mixture is converted into oxide, whilst the fluoride remains unchanged; the loss in weight is calculated to lanthanum acetate, lanthanum fluoride being found by difference. The results are stated to have been found satisfactory for quantities of fluorine of 0·001 to 0·14 grm.

W. R. S.

Estimation of Sulphur in Coal. H. Bahr. (*Chem. Zeit.*, 1924, **48**, 428.)—The reliability of Eschka's method is stated to be questionable in the case of coals rich in sulphur (over 2 per cent.). The method proposed is based on the aluminothermic reaction. A mixture of 0·5 grm. of coal, 0·4 of aluminium powder, and 3 of barium peroxide is briquetted; briquetting is indispensable to prevent loss by spirting. The briquette is ignited in a silica crucible covered with a perforated lid; all the sulphur present in the coal is converted into barium sulphide. After cooling, the crucible is treated in a suitable apparatus with dilute hydrochloric acid, and the hydrogen sulphide absorbed in cadmium solution, the sulphide being estimated with iodine and thiosulphate. The whole process may be carried out in 45 minutes. W. R. S.

Physical Methods, Apparatus, etc.

Application of Fluorescence Phenomena to the Identification of Various Drugs. E. Bayle and R. Fabre. (*J. Pharm. Chim.*, 1924, **29**, 535–543.)—The authors have examined the fluorescence emitted by certain crystalline organic bodies and solutions under the action of ultra-violet rays with defined conditions, using the Georges automatic lamp on a continuous current as the source of light, with a wave length of 3,650 A.U. By this means a differentiation of the constituents of crystalline mixtures, such as novocaine (violet fluorescence) and stovaine (no fluorescence), was easily effected, and the characteristic behaviour of a large number of organic bodies was studied, and in certain cases the length and intensity of the dominant wave length was noted. The results are recorded in a table, and it is shown that the methods employed are capable of many applications, such as, for example, the detection of impurities possessing different fluorescent properties from the substance containing them. Thus 1 part in 2000 of salicylic acid may be detected in milk, and the method will probably prove useful for identifying artificial colouring matters. (*Cf.* Kitching, ANALYST, 1922, **47**, 206.)

D. G. H.

Porous Porcelain Crucibles for Filtrations. G. F. Hüttig and K. Schmitz. (*Zeitsch. anal. Chem.*, 1924, **64**, 224–227.)—The Government porcelain manufactory of Berlin has made successful experiments in the manufacture of porous-bottomed porcelain crucibles to replace the Gooch crucible. They were tested as to their corrodibility, hygroscopicity, constancy in weight, etc., and found to satisfy the requirements of analytical practice. They cannot be used for the filtration of solutions of caustic alkalis, but this is not a serious drawback in ordinary analytical work. W. R. S.

Reviews.

CHEMICAL REACTIONS AND THEIR EQUATIONS. J. W. D. HACKLE. London: Chapman & Hall, Ltd. Pp. 138. Price 6s. net.

The author claims that this book is the only one of its kind, and in all probability, his claim is justified; the necessity for a volume devoted almost entirely to the construction and use of chemical equations is more open to doubt. There are many text books in use for class purposes which deal adequately with the subject, assuming, of course, that the teacher goes into the matter thoroughly. The book contains an assortment of information on chemical reactions, some of it very elementary in character and some considerably more advanced; by the time the student is in a position to appreciate the latter, the former should be quite superfluous. It is, therefore, difficult to recommend the book to either the beginner or the advanced student; the former could make little of such things as the ionic theory, and the latter should not need instruction on the preparation of simple salts and the adding up of molecular weights; furthermore, the classification of reactions into no fewer than twelve different types and the introduction of compounds of rare elements is surely unnecessary for either class of student. The volume closes with a thirty page appendix containing a displacement series of the elements, an outline of the Periodic System, a list of solubilities, and a glossary of chemical terms—a miscellany but little connected with what appears to be the general purpose of the book. A. F. KITCHING.

PHARMACEUTICAL AND FOOD ANALYSIS. By AZOR THURSTON. Pp. xiv.+416, with 19 Illustrations in Text. London: Chapman & Hall, Ltd. 1923. Price 21s. net.

The name of this book is truly stated above. The title page, but neither the cover nor the "jacket," gives a sub-title of "A Manual of Standard Methods for the Analysis of Oils, Fats and Waxes, and Substances in which they exist; together with Allied Products." The Preface announces that "a subsequent volume is being prepared to make the work a complete guide to the analysis of the more common drugs and foods."

The author has been for the past seventeen years chemist to the Ohio Agricultural Department, so the English reader has to make the best of American procedure in such directions as specific gravities taken at 25° C. (except in the case of alcohol). The drugs that by reason of fatty constituents get dealt with are those of the United States Pharmacopoeia, where camphorated oil and *Sapo Mollis* are made with cottonseed oil in place of our olive oil, where the official Oil of Cinnamon would be known here as the unofficial Oil of Cassia, and where Mercury Ointment is stronger than here. Such divergences are inevitable. In other respects, the author has been too diligent a student of English chemical books and journals to be unaware of English work, though many of the standards and other criteria used here would have been worth mention.

Our own journal is continuously mentioned in the Bibliography, but one reference is omitted: The four-figure alcohol tables in respect of their weight and volume values are those which Mr. Hehner compiled on the Fownes figure of 0·7938 for absolute alcohol. THE ANALYST celebrated the occasion by, for the first and last time, printing a serial; the Hehner tables were a monthly "feature" for five months in 1880. Mr. Thurston, of course, excludes the Proof Spirit column, but gives "grammes per 100 c.c." instead. The inclusion of this table does not mean that alcoholic liquids are dealt with—they are not—it is simply an appendage to a chapter dealing with twenty-five methods of taking specific gravities.

The author goes stolidly and, on the whole, effectively through the subjects indicated in the sub-title, and the chief failing is the omission of clauses here and there which would have protected the obtuse, the thoughtless and the ignorant, from gleaning wrong impressions. Page 217 reads: "Quite often dairymen determine milk acidity as lactic acid—10 c.c. of milk titrated with $N/10$ NaOH and the result multiplied by 0·9." Page 130 tell us "cocoa husks are roasted and used as cattle food"; a commentary to be found in THE ANALYST, 1920, p. 20, might well replace the statement. A cherry-red stain on "tumeric" (*sic*) paper is all the evidence the author needs that boric acid is present (p. 208), but when the reverse test, that for "tumeric" is required, the addition of alkali to the stain is necessary (p 215). The composition of human milk (p. 178), which occurs in a table of analyses of milk "from different animals," and is "compiled from various reliable authorities," includes percentages of fat as 5·61, and proteins as 1·27. Such peculiarities do certainly occur, as, for example, in an analysis by Richmond (ANALYST, 1908, p. 114), where the fat was 5·17 per cent. and the proteins 1·01 per cent., but it is a peculiar, and not a representative, specimen. The alkali treatment of cocoa is considered with no mention of the individual alkalis used. Treatment of sour milk with ammonia before analysis is advocated, with a footnote reference to THE ANALYST, 1885, 100. This was a paper by the late Dr. Adams, and was put forward as something preferable to a time allowance for decomposition, when the fat was being estimated by the Adams paper method. The experience of some others, when testing the method, has shown a few good results followed by many anomalous ones, and it seems to be little used in this country now.

In England we are accustomed to find figures for the average fat in cream to be, for example, 47·5 per cent. one year and 53·1 per cent. the next, but Mr. Thurston's analyses show an average of only 24·12 per cent., and the highest fat he records is only 35·21 per cent. (p. 237). However, the Federal standard of 18 per cent. explains much. On the other hand, the range of water in butter is from 5·99 per cent. to 12·59 per cent., and the wettest of twenty-four samples of margarine contained only 11·30 per cent. It is doubtful if any district in England can produce averages for water in either much below 15 per cent. The method given for the estimation of water in butter consists in drying two grms. "at a temperature of boiling water until a constant weight is obtained." There is no direction as to the time between the weighings from which constancy is taken for granted, but there is a *primâ facie* appearance, at any rate, of inadequate water removal.

To avoid gaps in his book, the author has made some analyses, but his readers have just cause for complaint against the person who supplied him with the sample of "cocoanut" (the spelling appears to be correct for America) oil which gave a Polenske number of 13·11 (p. 161). W. PARTRIDGE.

CHEMICAL SYNTHESIS. STUDIES IN THE INVESTIGATION OF NATURAL ORGANIC PRODUCTS. By HARRY HEPWORTH, D.Sc., F.I.C. Pp. xx. +243. London: Blackie & Son. 1924. Price 20s.

A very hearty welcome may be accorded to Dr. Hepworth's clear and concise summary of "the more important investigations which have been made by the organic chemist in modern times in the domain of natural organic products."

It is a well written book, and it certainly will do a great deal towards the dispelling of the impression held by some English chemists, that organic chemistry is mainly occupied in the making of organic compounds for Beilstein. There has certainly been a great deal of "beilsteinising" practised in recent years. However, Dr. Hepworth's summary of the classical investigations of Fischer (carbohydrates, purines and proteins), Willstätter (chlorophyll and anthocyanines) and Wallach (terpenes) leaves no doubt that organic chemistry still remains what it was meant to be: the true and honest chemical interpreter of life. It is for this reason that the reviewer was disappointed to find that Dr. Hepworth has devoted a whole chapter to the recent work on photosynthesis, most of which has been disproved, not only in this country, but also in America and Switzerland.

The book is fairly free from technical errors, although the names of some of the authors are wrongly spelt. Thus "Proctor" should read "Procter," "Freudenburg" "Freudenberg," "Zachmeister" "Zechmeister," etc., etc.

M. NIERENSTEIN.

DANGEROUS GOODS. By J. AEBY. Published by the author. Agents: Crosby Lockwood & Son, London. Pp. 320. 1922. Price 30s. net.

The author sets out to enumerate and briefly to describe all substances which may, for any reason, be described as dangerous merchandise from the point of view of shipping. Upwards of 450 different substances are mentioned, and, while it is obviously impossible to make such a work exhaustive, it is suggested that, if a substance is not mentioned in the book, it may be assumed that it presents no danger. Included in the term "dangerous" are substances which are either inflammable, explosive, corrosive, evil-smelling, or poisonous, and those which, although not dangerous in a strict sense, are liable to contaminate other merchandise.

The book is printed throughout in English, French and German, and there is a comprehensive index, also in the three languages, which gives both chemical and commercial names. For the most part, the merchandise mentioned comprises chemical products, but a number of important articles, such as brewers' grains, which are liable to heat spontaneously, or empty sacks which may be impregnated with nitrate, are mentioned on account of their practical importance. Attached to the name of each article is a brief description of its physical properties, in what way it is dangerous, and notes of any special precautions which are desirable in connection with it. With this information at hand it only remains for the shipper or his advisers to consider the physical state of the product, how it is packed and how it is stowed.

The printing and style of the volume are good, and, so far as the reviewer has observed, the information is accurate, save for a few obvious misprints. While containing few data which are not available to the chemist scattered in other works of reference, the book is sure to be a useful addition to the library of anyone—chemist or otherwise—who has occasion to advise on the subject of dangerous merchandise. H. E. Cox.

SUPPLEMENTARY NOTES ON GRAVIMETRIC ANALYSIS. W. LOWSON. Pp. 54. London: Longmans, Green & Co. 1923. Price 2s. 6d.

Although this is a small book of four chapters, it is filled with valuable information too frequently omitted from the ordinary text-book. Most of the points dealt with might be described as minor, but they are none the less of great importance when real accuracy is required. The title correctly indicates the scope of the work, which should prove useful to the advanced student and, in the more elementary portions, to the beginner also.

The first chapter deals with the choice of apparatus and the ordinary manipulative processes; and the second discusses the occlusion of soluble substances by precipitates; twenty pages are then devoted to notes on typical estimations—notes, the careful observance of which may make all the difference between poor and good results. After a short chapter on the calibration of apparatus, a list of solubilities is given, followed by a table of four figure logarithms. It is a book that can be thoroughly recommended to students. A. F. KITCHING.

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NOV.

THE ANALYST.

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held on Wednesday, October 1st, at the Chemical Society's Rooms, Burlington House. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Arthur Cecil Brooks, A.I.C.; William Donovan, M.Sc.; Albert George Flower, B.Sc.; Clifford Hollingsworth, F.I.C.; George Marshall Norman, B.Sc., A.R.C.S., F.I.C.; Charles Edward Sage, F.I.C.; Percy Frank Spendlove, B.Sc., A.R.C.S., A.I.C.; Charles William Spiers, M.Sc., A.I.C.; Leslie Hubert Trace, B.Sc., A.I.C.; and James Rawson Walmsley.

Certificates were read for the second time in favour of Messrs. Johan Ernst Nyrop, and Clifford Padgett Thorpe, B.Sc.

Messrs. G. W. Clough, D.Sc., J. P. Shenton, F.I.C., and W. S. Wood were elected members of the Society.

The following papers were read:—"The Estimation of Coconut Oil and Butter Fat in Margarine," by G. D. Elsdon and P. Smith; "A Preliminary Note on the Composition of the Fat of Goat's Butter," by F. Knowles and J. C. Urquhart; "The Quantitative Estimation of the Degree of Hydrolysis of Gallotannin by Tannase," by Miss W. N. Nicholson and D. Rhind; "The Pemberton-Neumann Method for the Estimation of Phosphorus," by Miss M. B. Richards and W. Godden and "Application of 'Formol Titration' to the Kjeldahl Method of Estimating Nitrogen," by W. S. Shaw.

Death.

It is with deep regret that we have to record the death, on September 9, of Mr. Otto Hehner, Past President, and one of the earliest members of our Society. An obituary notice will be published in the next issue.

The Recognition of Hydrogenated Oils.

BY K. A. WILLIAMS, B.SC., AND E. R. BOLTON, F.I.C.

(*Read at the Meeting, May 7, 1924.*)

THE test most widely applied for the detection of hydrogenated oils is based on the presence of traces of nickel in the oil, nickel being the catalyst used almost universally for the commercial hydrogenation of oils. The evidence given by this test is confirmed by the use of certain tests of minor importance and of doubtful reliability, the best known of which depends on the microscopic examination of the crystals deposited by the fat from its solution in ether.

The search for traces of catalyst is of assistance only when a positive result is obtained, since a negative result does not exclude the possibility of the presence of hydrogenated oil, and is of less value now than formerly owing to the greater skill of the manufacturer. in removing nickel from his hardened product. The trace of nickel which remains nowadays in these products is usually so small as to necessitate the use of a larger quantity of fat for the test than is usually available.

ISO-OLEIC ACID.—The authors have taken advantage, for the detection of hydrogenated oils, of a discovery first mentioned in 1913 by Lewkowitsch* and investigated by Moore.† These workers have pointed out that, during the course of hydrogenation of oils, there is formed an acid which had not previously been observed in oils derived from natural sources. This acid, which consists of one or more isomers of oleic acid, was termed iso-oleic acid, even though that name is used for other mixtures of oleic acid isomers.

The presence, then, of this new iso-oleic acid in edible fats in any quantity is proof of the presence of hydrogenated oils. It should be noticed, however, that Twitchell‡ states that he has found iso-oleic acid to be present to a limited extent (about one per cent.) in beef tallow. This appears to be the sole exception to the rule that iso-oleic acid does not occur in nature.

Iso-oleic acid appears to consist mainly of elaidic acid and contains probably certain other isomers of oleic acid, notably the 11.12 acid. It is a solid mixture at ordinary temperatures and has an iodine value of 90.

Its characteristic property is that, although unsaturated, it forms a lead salt nearly insoluble in most of the commonly employed organic solvents, *e.g.* ether, alcohol, benzene. In this it resembles the saturated acids, which also form insoluble lead salts, and differs from the other unsaturated acids whose lead salts are (except in the case of erucic acid and possibly of elaeostearic acid) soluble in these solvents.

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* Lewkowitsch, *Oils, Fats and Waxes*, I., 192 (5th Ed.).

† Moore, *J. Soc. Chem. Ind.*, 1919, **38**, 320T.

‡ *J. Ind. Eng. Chem.*, 1921, **13**, 806.

The estimation of iso-oleic acid is based on this property. The lead salts from a few grms. of the fat are formed quantitatively, and from these the lead salts of the unsaturated acids (*e.g.* oleic, linolic) are separated by extraction with one of the above-named solvents. The insoluble lead salts of the saturated acids and iso-oleic acid are washed as free as possible from the soluble lead salts. The acids are liberated from either or both fractions in ether; the ether is distilled off, and the acids are dried and weighed.

If the oil contains no hydrogenated oil the iodine value of the solid fraction will be not greater than 5 units (due to incomplete removal of unsaturated liquid acids in washing). If hydrogenated fat be present to any extent, this iodine value will be higher owing to the presence of iso-oleic acid, the content of which is calculated from this iodine value.

Allowing 5 units of iodine value in the solid fraction due to incomplete washing, the proportion of iso-oleic acid in the total fatty acids from the oil is given by the expression

$$\text{Percentage of iso-oleic acid} = \frac{95}{100} \times S \times \frac{I-5}{90}.$$

Where S = percentage of solid fraction obtained, and I = its iodine value.

The liquid fraction in each case contains the unsaponifiable matter of the fat. In a complete determination of the composition of the fatty acids this would have to be estimated and an allowance made for its effect on the iodine value of this fraction.* The solid fraction will be free from unsaponifiable matter, and, if only the proportion of iso-oleic acid is to be estimated, no correction is necessary.

SEPARATION OF SOLID AND LIQUID FATTY ACIDS.—Of the various methods of separation of solid and liquid fatty acids which have been proposed, the authors recommend as most satisfactory, either

(1) the lead salt and ether method of Gusserow-Varrentrapp; or

(2) the alcohol method of Twitchell.

Details of the procedure to be followed in the first of these may be found in most text books on oil analysis.† Details of the second are to be found in Twitchell's original paper.‡

Other methods the efficiency of which the authors have investigated include

(A) Separation of the thallium salts with water containing a small quantity of alcohol.§

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* Such determinations are described in full in "Fats, Natural and Synthetic," Middleton and Barry, p. 114.

† *E.g.* Bolton and Revis, *Fatty Foods*, p. 31.

‡ *J. Ind. Eng. Chem.*, 1921, **13**, 806, abstracted in ANALYST, 1921, **46**, 466.

§ Meigen and Neuberger, *Chem. Umschau*, 1922, **29**, 337.

(B) The separation of lead salts with

- (*a*) Petroleum spirit.
- (*b*) A mixture of 1 volume of petroleum spirit with 1 volume of methylated ether.

Using the first of these methods (A) and adhering to the authors' details, we have been quite unable to obtain an efficient separation. (*Cf.* ANALYST, **1924**, 448.)

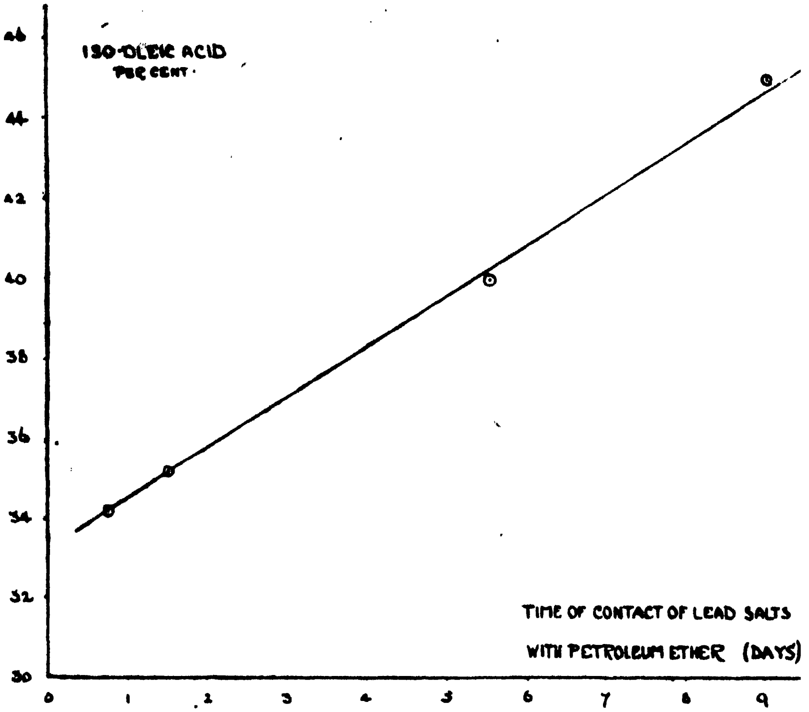

Fig. 1.

The results of separations by the other methods mentioned above on a typical hydrogenated cotton seed oil are shown in the table. It will be noticed that separation with mixed petroleum spirit and methylated ether gives results in close agreement with those obtained by the lead salt with ether or alcohol processes. Separation with petroleum spirit leads to erroneous results for the iso-oleic acid (and the oleic acid) content, the apparent iso-oleic acid content being much higher than that obtained by the use of any other solvent, and furthermore, not being constant, but varying with the time for which the lead salts are left in contact with the solvent. This variation is shown in Figure I. The shape of this curve appears to indicate that lead oleate crystallises slowly from petroleum spirit, even from dilute solution.

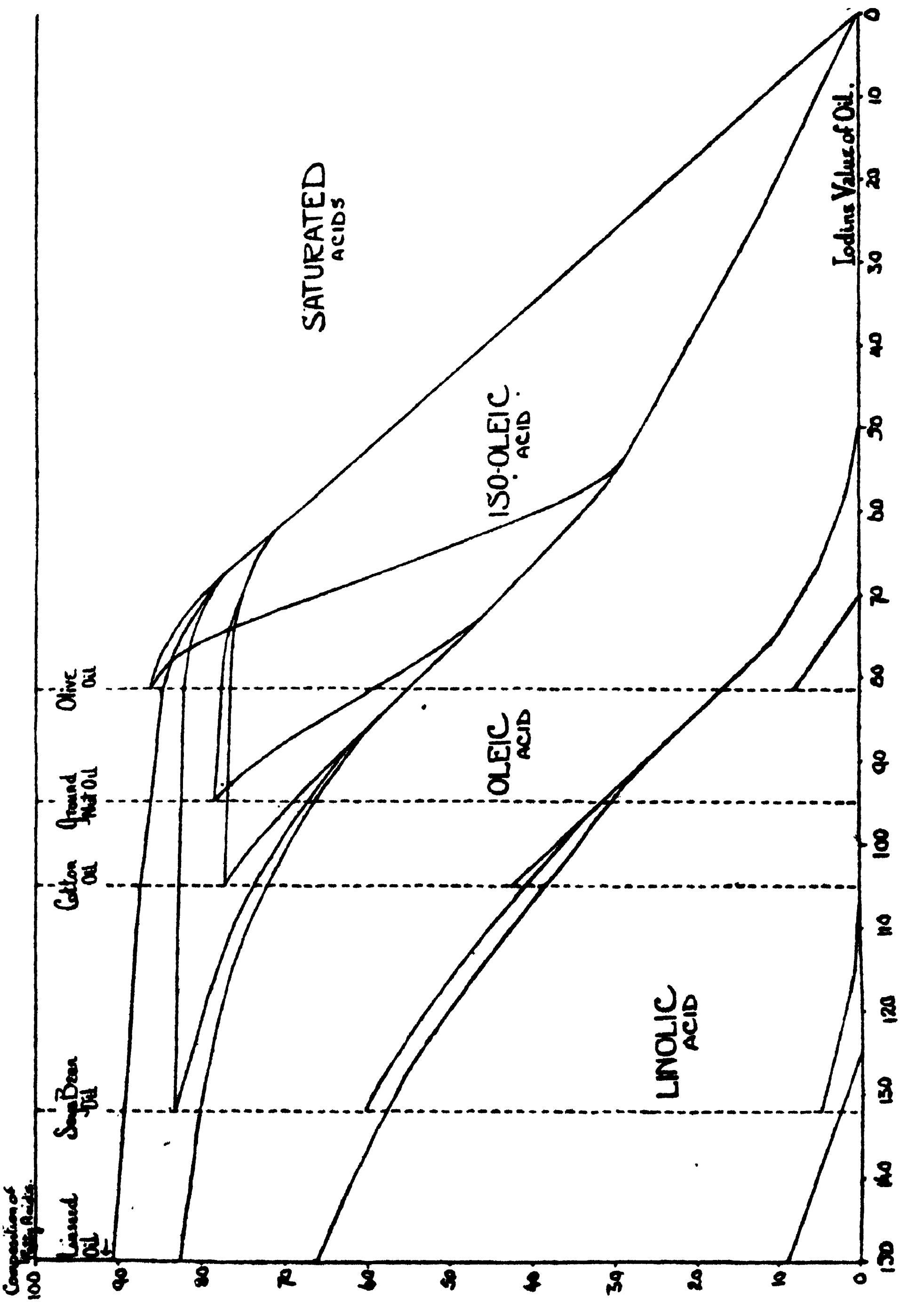
SATURATED ACIDS
ISO-OLEIC ACID
OLEIC ACID
LINOLIC ACID
Linseed Oil
Soya Bean Oil
Cotton Oil
Ground Nut Oil
Olive Oil
Composition of Fatty Acids.
Iodine Value of Oil.
100
90
80
70
60
50
40
30
20
10
0
0
10
20
30
40
50
60
70
80
90
100
110
120
130
140
150

Fig. 2.

HYDROGENATED COTTON SEED OIL.

Solvent used for separation of Lead Salts.	Time of contact of Lead Salts and Solvent.	COMPOSITION.			
		Linolic Acid.	Oleic Acid.	Iso-oleic Acid.	Saturated Acids.
Methylated ether	—	6·1	40·0	24·8	29·1
Alcohol	—	6·2	40·8	23·8	29·2
1 part meth. ether 1 part petroleum spirit	—	5·5	43·1	22·9	28·5
Petroleum spirit	18 hours	5·2	33·0	34·2	27·6
	1½ days	5·6	31·4	35·2	27·6
	5½ days	5·7	25·9	40·0	29·4
	9 days	5·2	21·7	45·0	28·1

APPROXIMATE ESTIMATION OF HYDROGENATED OIL.—As a guide to the approximate estimation of hydrogenated oil in mixtures we have prepared the curves in Fig. 2 which show the average compositions of the fatty acids from various hydrogenated vegetable oils at different stages of hydrogenation, the exact stage being shown by the iodine value of the fat.

These curves illustrate the well-known resemblance in composition between fats obtained by hydrogenation of different vegetable oils, and the consequent difficulty of determining the source of the hardened product.

Most commercial hydrogenated oils used in edible products have iodine values lying between 50 and 70 units and contain from 30 per cent. to 35 per cent. of iso-oleic acid, from which figures the approximate proportion of hydrogenated oil in the mixture may be deduced.

A drastic change in the composition of hydrogenated oils has recently resulted from a modification of the methods employed, by the introduction of a continuous process, whereby a flow of oil passes through a relatively large mass of catalyst.* The process can be operated so as to produce

(*a*) the normal proportion of iso-oleic acid in the product,
or (*b*) a reduced amount.

It will be obvious that the control of the proportion of iso-oleic acid on a commercial scale militates against the accuracy of the deductions made from the process the use of which the authors have suggested, with the result that in cases where the method of hydrogenation employed is unknown the proportion of hydrogenated oil may be largely over-estimated. The value of the process for the actual detection of hydrogenated oil remains, however, unaltered.

DISCUSSION.

Mr. W. H. SIMMONS, referring to Mr. Williams' remark that iso-oleic acid was discovered in 1913, said he thought that a perusal of the literature would probably prove that it had been discovered fifty or more years earlier. If, however, he was referring to a different isomer it was a pity the same name should be employed for two.

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* Eng. Pat. 162370, E. R. Bolton.

Mr. E. J. Lush suggested that the neutralisation value of the solid acids would be of assistance in deciding as to the oil from which a hydrogenated oil had been produced, as from this figure the proportion of palmitic acid in the oil could be obtained, this acid being one which remained constant in quantity during hydrogenation. The 50 iodine value point was interesting because it was the range used for soap and margarine. The composition chart gave the impression that when one got to an iodine value of 50 it was immaterial what oil one started from. He thought it would be very interesting to see a similar composition chart in which the melting point instead of the iodine value was used to denote degree of hydrogenation, because this would bring into play that important factor the tribasic nature of glycerin. Such a chart might bring out differences between different oils.

Mr. C. L. Hinton enquired whether corrections had to be made for the unsaturated acids not washed out from the saturated acids in the separation; and whether some corrections would not have to be made for unsaponifiable matter present in the fat.

Mr. K. A. Williams, in reply, said that, with regard to the corrections that would be applied normally to the iodine value, the iodine value of the solid acids obtained from an oil containing no iso-oleic was usually about 5, and it could be assumed that the unsaturated acids present as impurities in the solid acids would increase the iodine value of these by 5 units—*i.e.*, the iodine value obtained could be regarded as 5 units too high and be adjusted accordingly.

As to Mr. Lush's remarks he did not think that the neutralisation value of the solid fatty acids would help much, in that the solid acids of many of the oils were mostly palmitic acid, and the differences in the content of this acid in various liquid oils were not marked.

Regarding the question of composition and the tribasic nature of glycerin, there was scope for much work in this direction; the authors had not touched upon that point in the paper.

The Citric Acid Content of Milk Powder.

By DAN. W. STEUART, B.Sc. (Agr.).

(*Read at the Meeting, May* 7, 1924.)

Various estimations of the citric acid content of fresh milk which have been published indicate that from 0·15 to 0·2 per cent. is present. This would be concentrated some three-fold in the case of sweetened condensed milks, eight-fold in the case of full-cream milk powders, and eleven-fold in the case of skim-milk powders. The amount of citric acid in milk powder must then be quite considerable.

Two methods for estimating citric acid were tried, *viz.* by weighing it as pentabromacetone, and by weighing it as the double salt of mercury acetone dicarboxylate with basic mercury sulphate.

A. STAHRE'S PENTABROMACETONE METHOD.*—Five grms. of the milk powder were dissolved in 45 c.c. of warm water, 10 cc. of 50 per cent. sulphuric acid added, and then 2 c.c. of 40 per cent. potassium bromide solution, and 20 c.c. of 10 per cent. phosphotungstic acid. After mixing, the whole was filtered, and the filter was washed with water till the filtrate measured 150 c.c. To this was added excess of fresh bromine water, and the temperature raised to 50° C. for 5 minutes. Then potassium permanganate solution (5 per cent.) was added, drop by drop, slowly, so that the temperature did not rise appreciably, and with stirring all the time, till a brown precipitate formed. Ferrous sulphate solution was now stirred in till the precipitate was decolorised. After standing overnight, the precipitate was collected on a prepared asbestos Gooch filter, washed with water acidified with sulphuric acid, and with distilled water, and then dried for 24 hours in a vacuum desiccator over sulphuric acid.

Weight of precipitate × 0·424 = Anhydrous citric acid.
" " " × 0·464 = Crystalline citric acid.

The following percentages of anhydrous citric acid in milk powders were obtained.

Full cream, per cent.:

1·11, 1·13, 1·21, 0·98, 1·30, 1·13, 0·97, 1·31, 0·96, 1·31, 1·21, 1·27, 1·17.

Skim-milk, per cent.:

$\left.\begin{matrix}1{\cdot}48\\1{\cdot}50\end{matrix}\right\}$ av. 1·49; $\left.\begin{matrix}1{\cdot}65\\1{\cdot}80\end{matrix}\right\}$ av. 1·72; $\left.\begin{matrix}1{\cdot}37\\1{\cdot}58\end{matrix}\right\}$ av. 1·47; 1·55, 1·73, 1·46, 1·68, 1·39, 1·60, 1·47, 1·60.

The precipitate consisted of practically pure penta-bromacetone. A trace of brominated protein was removed by dissolving the substance in ether and filtering the solution. The recovered compound melted at 72 to 75° C. and yielded 88·0 per cent. of bromine (by Stephanow's method followed by titration by Volhard's method).

Pure crystalline citric acid was then used for a series of estimations after verifying the purity by the neutralisation value and the moisture content. Seven estimations, in which 0·1 grm. in 150 c.c. of water was used, gave from 90·0 to 93·5 per cent. (av. 92 per cent.) of the theoretical yield of pentabromacetone. The low results were attributed to over-oxidation.

B. BEAU'S MERCURY SULPHATE METHOD.†—In this case 5 grms. of milk powder were transferred to a 200 c.c. flask with 100 c.c. of water. Then 50 c.c. of mercury sulphate reagent were added and a little 10 per cent. phosphotungstic acid. After mixing, the solution was made up to the mark and filtered. To 100 c.c. of the filtrate at the boiling point, 1 per cent. potassium permanganate solution was added, drop by drop, with constant stirring, till a brown precipitate settled down. The liquid was boiled again, and a little hydrogen peroxide solution stirred in till the precipitate looked clean. The precipitate was then collected on a Gooch filter, washed with water and dried at 105° C. for 2 hours.

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* Abstracts in ANALYST, 1922, 47, 24; 1916, 41, 378.

† See Local Govt. reports on Milk Powders, 1920; also Barthel's "Milk and Dairy Products."

Weight of precipitate × 0·271 = Anhydrous citric acid.
„ „ „ × 0·296 = Crystalline citric acid.

The usual method of preparing the mercury reagent is as follows:—To 50 grms. of red oxide of mercury and 400 to 500 c.c. of water in a litre flask add sufficient (about 75 c.c.) concentrated sulphuric acid, gradually, to dissolve all the powder. Make up the solution to 1000 c.c., boil and filter. This solution gave too high results when pure citric acid was used. The Local Govt. Report states that 5 c.c. of the reagent should require 7 to 8 c.c. of *N* sodium hydroxide solution before giving a permanent turbidity. The reagent was thus considered to be insufficiently acid. When this was put right the results obtained with pure citric acid were low. The following percentages of anhydrous citric acid in milk powders were obtained:

Full cream, 1·21, 1·13, 0·89, 1·20, $\left.\begin{matrix}1{\cdot}42\\1{\cdot}44\end{matrix}\right\}$ av. 1·43.

Skim milk, 1·42.

The precipitate in this case was filtered off with greater difficulty than in the case of the former method.

SUMMARY.—The amount of citric acid in milk powder is quite considerable, being equal roughly to one-fifth of the ash. A sample of fresh milk (April) gave 0·158 per cent. of anhydrous citric acid. The average figure obtained with full cream milk powders was 1·16 per cent., and with skim milk powders 1·55 per cent. In each case this would be equal to 0·144 per cent. in the original milk. These figures are on the low side.

An Apparently Specific Test for Tannins.

BY ALAN H. WARE.

To the present writer the term "tannin" connotes a complex phenol, or phenolcarboxylic acid, which is astringent to the taste, water-soluble, will convert hide or skin into leather, and form an ink with soluble simple iron salts. The goldbeater's skin test for tannins (ANALYST, 1924, 25) has a special value in that it recognises, and combines in one concrete form both the chemical reactions named in the above definition. For research work, and for use in checking the validity of any new test, either the goldbeater's skin test or some similar test, or set of tests, will always be necessary.

As a routine test, however, the goldbeater's skin test suffers from the following disadvantages, *viz.* (1) That it takes fully an hour to complete, and this is a very long time for a routine qualitative test. (2) That, whilst a negative result undoubtedly implies the absence of tannin, a positive result, with respect to iron-coloration, does not *necessarily* imply the presence of tannin; for certain other substances give iron-colorations on the skin closely resembling the results yielded by weak tannin extractives. Hitherto no single test has been published for tannins *as a class*, which answers the several and combined requirements of being really specific, suitable for rapid routine work, and capable of giving obvious and emphatic results. The writer believes that the test here described will fulfil all these desiderata.

The test has been evolved from a consideration of Mitchell's ferrous tartrate test (ANALYST, 48, 1923, 1). This test was devised for quantitative work, and Mitchell, of course, makes no claim that it is specific for tannins, seeing that he uses it also for estimating gallic acid. Further, the test, as it stands, gives colour-reactions with a number of phenolic substances, more especially if tap-water is used. It seemed, however, to the present writer that this test indicated possibilities worth investigating. Its use, of course, depends largely upon the well-known fact that iron is not precipitated from solution by commonly used reagents in the presence of tartrates. Mitchell used Rochelle salt and ferrous sulphate, and found that, with this reagent, tannin would not precipitate the iron, but that a bluish-violet coloration was obtained the intensity of which was related to the amount of gallic acid or gallotannin present.

It occurred to the writer that the test might possibly be so modified as to precipitate all tannins and no other water-soluble substances found in plant extractives. He did not quite succeed in obtaining this result with Mitchell's reagent, but by experimenting with ferric salts and citrates, he ultimately found that by using the iron and ammonium citrate of the British Pharmacopœia, together with ammonium acetate, an almost ideal reagent was obtained. A full list of results is given below, but for the moment it may be said that, with the exception of hæmatoxylin, the writer has so far been unable to find any other chromogenic water-soluble phenols which are precipitated by the method here described for the precipitation of tannins. Hæmatoxylin is readily distinguished, because it is only partially precipitated and yields a deep blue filtrate. Certain distinctive results were also obtained with several other phenolic substances, and will be described. By means of this test gallotannins can be readily and quickly distinguished and separated from gallic acid, and catechu tannins from catechins. Catechol tannins also can be quickly distinguished from such bodies as iron-greening anthoxanthins, caffeo-tannic acid (so-called) and ipecacuanhic acid.

METHOD OF TESTING.—A solution of 0·25 grm. of iron and ammonium citrate in 100 c.c. of tap water is prepared. The iron compound (?) is often slightly acid, and if this is not rectified by the alkalinity of the tap-water, the solution should be made *very* slightly alkaline by the cautious addition of aqueous ammonia. It is important that the iron citrate should answer the pharmacopœial requirements as to general characters and purity, more especially as there is a green citrate, also sometimes used in medicine, which on no account must be used for this test.

The actual method of applying the test depends upon the nature of the extractive and the purpose of the test. Preliminary experiments seem to indicate that the test may be advantageously used in certain cases as a quantitative method. The present paper, however, deals only with the qualitative use, in which two principal cases arise:

(1) If a freshly made, clear, aqueous extractive or solution has to be tested, place 4 c.c. of this in a test-tube, and add, successively, 4 c.c. each of the iron solution and of a 30 per cent. solution of ammonium acetate. Shake well. Note the colour or appearance, and then boil. Filter. Complete reaction should be ensured by testing a portion of the filtrate with more of the ferric citrate solution and observing if there is any further change. Note colour of precipitate (if any) and of filtrate or solution. Boil the latter with a little 10 per cent. solution of ammonia and note whether there is any further change. The precipitate should be insoluble in solution of ammonia or dilute acetic acid, but is soluble in dilute hydrochloric acid.

(2) If testing a pharmaceutical or other ready-made extractive which appears to be an alcoholic or concentrated aqueous preparation, it is advisable to proceed as follows:—Dilute sufficiently with 10 per cent. solution of ammonium acetate. Warm. Filter. Wash precipitate with successive small quantities of boiling water to dissolve salted-out tannin, and collect the washings with previous filtrate. Add the iron reagent and proceed as before. By this method resin, fat, chlorophyll and other substances which are likely to interfere with the success of the test are sufficiently removed. Some of the less soluble phlobaphenes, in the case of phlobotannin drugs, are also lost, but this is not a disadvantage, as these are scarcely to be regarded as true tannins.

STATEMENT OF RESULTS.—A very large number of pure substances and vegetable bodies have been tested, and some of the results may be summarised thus:

(1) True tannins appear to be completely precipitated from all substances known to contain them. With gallotannins a purplish precipitation commences in the cold and is complete on boiling. Without the ammonium acetate there is neither precipitation nor purple coloration. Indeed, if the iron reagent is boiled with the tannin solution before adding the ammonium acetate, subsequent precipitation becomes difficult or incomplete.

(2) The only other substance which has yielded a precipitate under the conditions named for tannins is hæmatoxylin, and this can be readily distinguished from tannin by giving a deep blue solution and no purplish precipitate in the cold. Further, on boiling with excess of the reagent partial precipitation, only, takes place, a blue-black precipitate and a deep blue filtrate being yielded. Logwood in fresh decoction gives a similar result.

(3) Catechol, protocatechuic acid, catechins, pyrogallol, gallic acid and brazilin (in the form of decoction of sappan wood), all give purplish-violet solutions on boiling. On the addition of solution of ammonia the colour changes to an intense reddish tint. There is no precipitate in any case.

(4) Carbolic and salicylic acids, resorcinol, hydroquinone, phloroglucinol, and anthraquinone give no characteristic result until the solution of ammonia is added, when there is some intensification of the brown colour.

(5) Quercetin, rutin, aloin, and, as far as can be judged from extractives containing them, all other water-soluble anthoxanthin and oxymethyl-anthraquinone bodies give a brown coloration which is considerably intensified on adding solution of ammonia. This result is also yielded by *Nux vomica* (caffeo-tannic acid, so-called), by hops (so-called hop-tannin, etc.) and probably by the majority of plant extractives which do not contain tannin. A certain number of extractives containing anthoxanthins (? flavones), if sufficiently strong, yield a precipitate *after* the addition of ammonia and reboiling, notably those of *Reseda luteola*, *Digitalis* leaves and *Stramonium* leaves. *Viburnum prunifolium* and cotton root barks appear to give a similar result. Alcoholic extractives treated by the second method generally answer best. This test distinguishes a reliable pharmacopœial tincture of *digitalis* made with properly collected and dried leaves from many other chlorophyll-containing tinctures, *e.g.* those of belladonna and hyoscyamus.

(6) Anthocyanin extractives which contain no tannin and little or no anthoxanthin, *e.g* blue cornflower and poppy petals in decoction, give no very characteristic result.

THE QUALITATIVE SEPARATION OF PHLOBOTANNIN, GALLOTANNIN AND GALLIC ACID IN SOLUTION.

This may be readily effected by combining Stiasny's test, as modified and extended by the present writer (ANALYST, 1924, 442), with the test described above.

METHOD.—Boil 10 c.c. of aqueous solution or extractive (diluted with water if alcoholic) with excess of 10 per cent. hydrochloric acid and 40 per cent. formaldehyde (12 drops of each are usually ample) for one minute. Cool and filter. The precipitate will contain all the phlobotannin present. The short period of boiling does not result in any very appreciable quantity of other phenolic bodies being precipitated. Therefore, if, after washing successively with water, 90 per cent. alcohol and 10 per cent. solution of ammonia, there is a residue left which is very obvious with respect to colour and quantity, it almost certainly indicates phlobotannin. Confirm by testing with bromine water or by any other suitable test applied to the original solution or extractive.

The filtrate from the formaldehyde test may be divided into two parts, one of which should be tested as described in the paper cited above for iron-greening or iron-blueing phenolic bodies. If iron-blueing occurs, the second portion should be neutralised by the cautious addition of a weak solution of ammonia, and tested by boiling with the ammonium ferric citrate and ammonium acetate solutions as described in the present paper. A purple or violet precipitate is due to gallotannin. If gallic acid be present, the filtrate (or solution, if gallotannin be absent) will be purplish-violet. In the event of the gallic acid content being small, the reddish colour which may be obtained by adding solution of ammonia is often more intense than the previously existing violet colour.

SUMMARY AND CONCLUSIONS.—A new test for tannins and certain other chromogenic phenols is described, the reagent used being the iron and ammonium citrate of the British Pharmacopœia together with ammonium acetate. Very distinctive results are obtained with tannins and hæmatoxylin. These results differ from one another and from those yielded by any other water-soluble substances known to the writer. Tannin alone is completely precipitated by the method employed. Hæmatoxylin is incompletely precipitated. No other water-soluble phenols or phenolic glucosides appear to yield precipitates in neutral solution, but a purplish-violet coloration is given by catechol, protocatechuic acid, catechin, pyrogallol, gallic acid and brazilin. This colour is changed to an intense reddish colour on the addition of ammonia. Vegetable extractives containing phenolic bodies other than those mentioned give an intense brown colouration, more especially after the addition of ammonia. Caffeo-tannic acid, so-called and the reputed tannin of hops are not precipitated. All the plant extractives which yield a precipitate in this test, give a marked response to the goldbeater's skin test and also give positive results with the modified and extended Stiasny's test, described elsewhere by the present writer. The test appears, therefore, to be quite specific for tannins, and also yields valuable results with certain other phenolic substances. It is of especial value in pharmacognosy, and plant analysis generally, and may prove to be useful for quantitative purposes.

UNIVERSITY COLLEGE,
EXETER.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

PUBLIC HEALTH (DRIED MILK) REGULATIONS, 1923.

CALCULATION OF "EQUIVALENT PINTS."

THE "equivalent pints" of a quantity of dried milk are given by the following formulæ, similar to those previously published (ANALYST, 1923, **48**, 596) for condensed milk.

W. is the total weight of dried milk in grms., and F., T.M.S., and S.N.F. are the percentages by weight of fat, total milk solids and solids-not-fat, respectively, in the dried milk.

Milk.

$$\text{Equivalent pints} = \frac{\text{F.}\times\text{W.}}{2107} \quad \text{or} \quad \frac{\text{T.M.S.}\times\text{W.}}{7258}.$$

Three quarter cream milk.

$$\text{Equivalent pints} = \frac{\text{F.}\times\text{W.}}{1582} \quad \text{or} \quad \frac{\text{T.M.S.}\times\text{W.}}{6797}.$$

Half cream milk.

$$\text{Equivalent pints} = \frac{\text{F.}\times\text{W.}}{1056} \quad \text{or} \quad \frac{\text{T.M.S.}\times\text{W.}}{6335}.$$

Quarter cream milk.

$$\text{Equivalent pints} = \frac{\text{F.}\times\text{W.}}{528} \quad \text{or} \quad \frac{\text{T.M.S.}\times\text{W.}}{5811}.$$

Skimmed milk.

$$\text{Equivalent pints} = \frac{\text{S.N.F.}\times\text{W.}}{5285}$$

E. HINKS.

CALCULATIONS: PUBLIC HEALTH (DRIED MILK) REGULATIONS, 1923.

UNDER the above Regulations it is necessary to calculate the number of pints of milk of stated composition which are equivalent to the contents of a tin of dried milk.

		Dried Milk.			
		Milk.	¾ Cream.	½ Cream.	¼ Cream.
Composition of corresponding standard milk	Fat	3·6	2·7	1·8	0·9
	T.S.	12·4	11·6	10·8	9·9
Minimum limit of fat per cent. ..		26·0	20·0	14·0	8·0
Sp. Gr. of standard milk		1·032	1·0328	1·034	1·0347
Formulæ, for calculation from T.S. found of equivalent milk in ounces		$\frac{\text{C.}\times\text{T.S.}}{362\cdot 7}$	$\frac{\text{C.}\times\text{T.S.}}{339\cdot 6}$	$\frac{\text{C.}\times\text{T.S.}}{316\cdot 6}$	$\frac{\text{C.}\times\text{T.S.}}{290\cdot 4}$
Formulæ, for calculation from fat found of equivalent milk in ounces		$\frac{\text{C.}\times\text{F.}}{105\cdot 3}$	$\frac{\text{C.}\times\text{F.}}{79\cdot 06}$	$\frac{\text{C.}\times\text{F.}}{52\cdot 76}$	$\frac{\text{C.}\times\text{F.}}{26\cdot 40}$
Per cent. of fat when dry "dried milk" of minimum limit fat content is diluted to contain the prescribed total solids		3·22	2·32	1·51	0·79

C = Weight of dried milk in tin in grms.

It is evident from the above figures that it is impossible to dilute dry "dried milk" of minimum limit fat content to milk of standard composition.

The following table shows the composition of each variety of dried milk containing the minimum percentage of fat, which when diluted to the volume calculated from the T.S. would produce standard milk.

		Milk.	¾ Milk.	½ Milk.	¼ Milk.
Water	..	10·44	14·08	16·00	12·00
Fat	..	26·00	20·00	14·00	8·00
S.N.F.	..	63·56	65·92	70·00	80·00

Below are tabulated the percentages of fat corresponding to percentages of moisture in each variety of dried milk, which when diluted to the volume calculated from the total solids will yield the standard milk.

	Per Cent. Fat.			
Per cent. Moisture.	Milk.	¾ Milk.	½ Milk.	¼ Milk.
0	29·03	23·28	16·67	9·09
1	28·74	23·04	16·50	9·00
2	28·45	22·81	16·33	8·91
3	28·16	22·58	16·17	8·82
4	27·87	22·35	16·00	8·73
5	27·58	22·11	15·83	8·64
6	27·29	21·88	15·67	8·55
7	27·00	21·65	15·50	8·45
8	26·71	21·41	15·33	8·36
9	26·42	21·18	15·17	8·27
10·44	26·00	—	—	—
12·00	—	—	—	8·00
14·08	—	20·00	—	—
16·00	—	—	14·00	—

The table is used for calculation thus:

If the "dried milk" contains the same or more fat than the figure given in the table to correspond with the moisture found, the dilution is calculated from the total solids and will give a milk of standard composition as regards total solids with a fat equivalent to or more than the standard. If the milk contains less than the fat corresponding to moisture found, the dilution is calculated from the fat, and the resulting milk will contain the standard amount of fat with an excess of total solids.

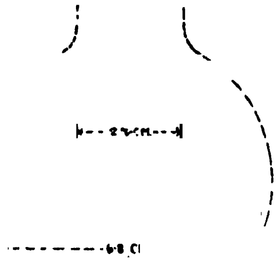
DOUGLAS HENVILLE.

ERRATUM.

CONDENSED MILK REGULATIONS, 1923.

ANALYST, 1923, **48**, 597, line 13 from bottom: *For* "total milk solids in the tin" *read* "total milk solids in one pint."

NEW FLASK FOR USE WITH SOXHLET'S APPARATUS.



THE flask employed with the Soxhlet apparatus is commonly unnecessarily large and heavy. The flask illustrated has been made for the writer and found very satisfactory in practice. It is made of thin glass and is very light in weight. In addition to giving increased accuracy, the distillation is accomplished with the minimum of heat and the extract is dried with greater rapidity.

ROBERT C. FREDERICK.

ROYAL NAVAL MEDICAL SCHOOL,
ROYAL NAVAL COLLEGE, GREENWICH.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY AND COUNTY OF KINGSTON-UPON-HULL.

REPORT OF THE PUBLIC ANALYST AND BACTERIOLOGIST FOR 1923.

DURING the year 1068 samples of foods and drugs were examined, of which 538 were purchased informally. Of the total number, 58 were reported as adulterated and 33 as suspicious, the percentage of the two being 8·5, as compared with 12·0 in 1922. The number of samples per 100,000 of the population was 372, as compared with 300 for England and Wales during 1921.

MILK.—Of the 436 samples examined, 23 were adulterated and 23 suspicious. Eleven samples contained an unwarranted amount of "dirty" sediment, and 6 were returned as adulterated for this reason. Proceedings were taken against a farmer who had supplied 3 of these "dirty" milks. These samples were proved in court to be seriously contaminated with dung, and the Corporation won an appeal in the High Court on the method of sampling adopted, but the local magistrates finally dismissed the summons on the ground that there was no legal standard for the proportion of extraneous sediment in milk.

DRIPPING.—Two samples (from the same shop), out of 13 examined, were adulterated. They contained about 14 per cent. of water, 1 per cent. of salt, and 3·6 per cent. of non-fatty organic impurities (flour, etc.). Proceedings were taken in the case of one of these samples, and evidence was given that dripping should not contain more than small amounts (up to 2 per cent.) of water and other extraneous matters; this definition was in agreement with the Ministry of Health standard for dripping during the war. The justices, however, decided that the substance sold was "dripping," and dismissed the case.

Flour and Bread.—Of a total of 10 samples of flour, 4 contained potassium persulphate, a so-called "improver," and 3 of the 4 were also slightly bleached with nitrogen peroxide. In the present state of the law this treatment of flour cannot be stopped.

Farinaceous Foods.—Four samples of pearl barley and 3 of arrowroot were adulterated. The pearl barley contained about 1 per cent. of rice flour as a "facing" over the grains of barley. The adulterated arrowroot samples consisted of cream of tartar (1) and maize flour (cornflour) (2). They were probably all accidental substitutions.

Canned Foods.—Three of 6 samples of canned tomatoes contained 5·2, 3·1 and 1·9 grains of tin, respectively. All came from one vendor, who was convicted.

Drugs.—Of 158 samples examined, 9 were reported to be adulterated. These were:—Almond oil (2), which consisted largely of a foreign kernel oil; milk of sulphur (2), containing about 37 per cent. of hydrated calcium sulphate; milk of sulphur (1), containing 25 parts of arsenious oxide per million; sweet spirit of nitre (2), deficient to the extent of 40 and 42·8 per cent. in ethyl nitrite; tincture of quinine (1), deficient 10 per cent. in alcohol; and turpentine oil, which consisted wholly of a petroleum distillate.

Bacteriological Examinations.—In all, 3,744 specimens were examined, 3,733 of which were pathological material, and 11 were samples of water.

A. R. Tankard.

CITY OF BIRMINGHAM.

Annual Report of the City Analyst for 1923.

Of the 5,197 samples analysed during the year, 4,518 were taken under the Sale of Food and Drugs Acts, and of these, 3,939 samples were bought informally. With the exception of milk, samples were rarely bought formally unless a previous informal sample had been found to be adulterated. The number of samples purchased was at the rate of 483 for every 100,000 persons living in the city.

Milk.—During the year 2,441 samples of milk were analysed, being at the rate of 261 per 100,000 persons, and, of these, 158 were adulterated.

Condensed Milk.—The Regulations appear to be defective in making no distinction between the composition of condensed skimmed milk and condensed machine-skimmed milk. One of the samples received was marked "skimmed" on the declaration, and "machine-skimmed" on another part of the label. As it only contained 0·4 per cent. of milk fat, the declaration was incorrect, as *uncondensed* skimmed milk may contain 1 per cent. of fat. The vendor of the sample was cautioned. Five other samples examined practically complied with the Regulations.

Butter.—Seventy-six per cent. of the samples were free from boric acid, and in the remainder the quantity did not exceed 0·5 per cent.

Golden Syrup.—Only 3 of 9 samples were genuine, the others containing from 25 to 90 per cent. of glucose syrup. A circular was sent to members of the grocery trade calling attention to the necessity of mixtures of golden syrup and glucose syrup being suitably labelled and not sold as "golden syrup."

DRUGS.—Twenty-six of the 218 samples examined were unsatisfactory.

Camphorated Oil.—Four samples were practically correct (19·7 to 20·2 per cent. of camphor), but two from one vendor contained 13·7 per cent. and 14·9 per cent., and the vendor was prosecuted and fined.

Boric Acid Powder.—Nine samples contained from 4 to 24 parts of lead per million, and one sample contained 80 parts, for the sale of which the vendor was cautioned.

Potassium Carbonate.—Two samples from one vendor contained about 80 parts of lead, and 7 and 10 parts of arsenic per million, with 20 per cent. and 17·9 per cent. of moisture, respectively. The vendor was prosecuted and it was proved that when the drug was sent to the shop it complied with the Pharmacopoeial requirements, and the explanation given was that the drug had dissolved lead and arsenic from the shop bottle. The magistrates dismissed the case on the ground that the drug was "unavoidably mixed with some extraneous matter in the process of collection or preparation," and that "collection" included storage (*cf.* ANALYST, 1923, 48, 215, 260). Experiments made subsequently proved that the explanation was correct (*cf.* ANALYST, 1923, 48, 543).

J. F. LIVERSEEGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

SALE OF "BUTTER MIXTURE."

ON August 13th a dairyman was summoned at Salford for selling an article not of the nature, substance and quality demanded, and an Irish firm of margarine manufacturers was summoned for aiding and abetting.

Mr. R. H. Wright, for the prosecution, said that the inspector sent a messenger for half a pound of "butter mixture"—not margarine. The analysis showed the sample to contain only 1 part of butter in 200, so that, whatever "butter mixture" might mean, this sample could not be said to be butter mixture. The article was supplied by the defendant company and was accompanied by advertisement slips referring to it as "butter mixture."

Mr. Sandbach, for the defence, submitted that, in view of rulings, the firm had a right to describe the article as "butter mixture" if they wished; but, as the Stipendary had recently given a decision on the point, they did not intend to exercise that right, and had withdrawn altogether the description "butter mixture," so as to fall into line with the Stipendiary's view of the law. According to a decision in the King's Bench Division his clients had a right to describe a mixture of margarine and butter—no matter what the proportions were—as "butter mixture," but they had no wish to do so.

The Stipendiary said that as the offence was practically admitted, the defendant would be fined 20s. and the manufacturers 40s.

A second summons for selling the sample in a wrapper having on it a descriptive name other than the word "margarine," was dismissed, as the Stipendiary considered that the use of the fancy name was covered by the decision in *Hawes* v. *Stephens*, but fines were imposed for the use of a wrapper bearing the word "margarine" otherwise than in ½ inch block letters.

Tests on Volumetric Glassware. Classes A & B.

THE NATIONAL PHYSICAL LABORATORY, TEDDINGTON, MIDDLESEX. METROLOGY DEPARTMENT. SEPT. 1924.[*]

INSTRUCTIONS as to transmission, insurance and custody of apparatus, are followed by regulations governing the two classes of tests.

UNITS OF VOLUME.

(1) *The Litre.*

The litre is defined as the volume occupied by one kilogramme of water at its temperature of maximum density and subjected to normal atmospheric pressure.

(2) *The Cubic Centimetre.*[†]

This is the volume occupied by a cube each of whose edges is one centimetre in length.

The accepted relation between the cubic centimetre and the millilitre is

1000 ml. = 1000·027 c.c.

(3) *British Imperial Units.*

The gallon is defined as the volume occupied by ten imperial pounds weight of distilled water as weighed in air against brass weights, with both the water and the air at a temperature of 62° F. and the barometer at 30 inches.

The gallon has now been legalised as 4·5459631 litres.
The cubic inch is equivalent to 16·38659 ml.

(4) *The G.W.A. Unit.*

This is defined by the relation 1000 G.W.A. = 1002 ml.

Marks.

№.A. ’24 №.B. ’24 respectively denote that the apparatus is correct within the specified tolerance and certificates are issued for Class A where required.

TOLERANCES.

Flasks.

	Capacity ml.	10	25	50	100	250	500	1000	2000
A	Tolerance ± ml. for content	0·015	0·03	0·04	0·06	0·1	0·15	0·2	0·4
B	Tolerance ± ml. for content	0·02	0·04	0·06	0·1	0·2	0·3	0·4	0·6
	for delivery ..	—	—	—	0·2	0·4	0·6	0·8	1·0

NOTE.—(*a*) For capacities not tabulated, the tolerance is that of the next larger tabulated capacity.

Pipettes.

	Capacity ml.	2	5	10	20	30	50	100	250
A	Tolerance ± ml. for content	0·01	—	—	0·02	0·03	0·04	0·06	0·10
B	delivery	0·02	0·03	0·04	0·05	0·06	0·08	0·12	0·2

[*] The Director of the National Physical Laboratory, Teddington, Middlesex, has kindly offered to supply members of this Society with a copy of this pamphlet, free of charge, on application being made to him.—EDITOR.

[†] The use of the millilitre to express the capacity of volumetric glassware is strongly recommended.

Graduated Pipettes.

	Total capacity ml.	2	5	10	30	50	100
A	Maximum error allowed at any point tested and also maximum difference allowed between the errors at any two points tested for content or delivery ± ml.	0·01	0·02	0·03	0·06	0·1	0·15
B	Maximum error allowed at any point tested and also maximum difference allowed between the errors at any two points tested ± ml.	0·02	0·04	0·06	0·1	0·2	0·3

See Notes (*a*) and (*b*) under Burettes.

Burettes.

	Total capacity ml.	2	5	10	30	50	100	200
A	Maximum error allowed at any point tested and also maximum difference allowed between the errors at any two points tested for content or delivery ± ml. ..	0·01	—	0·02	0·03	0·06	0·1	0·2
B	Maximum error allowed at any point tested and also maximum difference allowed between the errors at any two points tested ± ml.	0·02	0·03	0·04	0·06	0·01	0·02	0·4

NOTE.—(*a*) For capacities not tabulated, the tolerances are the same as those given for the next larger tabulated capacity.

(*b*) The tolerances apply to the whole of the graduated portion and to any fraction of it. Thus for example, a 50 ml. burette may be in error by ±0·06 ml. (Class A) or ±0·1 ml. (Class B) at any point provided that the difference between the errors at any two points tested does not exceed 0·06 ml. (Class A) or 0·1 ml. (Class B).

Graduated Cylinders.

	Total capacity ml.	5	10	25	50	100	250	500	1000	2000
A	Maximum error allowed at any point tested, and also maximum difference allowed between the errors at any two points tested: for content ± ml. ..	0·04	0·06	0·1	0·2	0·4	0·6	1	2	3
B	,, ,,	0·06	0·1	0·2	0·4	0·8	1·5	2	4	8

NOTE.—The notes (*a*) and (*b*) relating to tolerances on burettes (*supra*) apply also to graduated cylinders.

Specific Gravity Bottles or Tubes.

	Capacity ml. ..	10	25	50	75	100	150	200	250
A	Tolerance ± ml. ..	0·003	0·005	0·008	0·010	0·012	0·015	0·020	0·025

Gas Analysis Vessels.
measuring bulbs.
pipettes.
Measuring tubes, gas burettes, eudiometers, nitrometers, pressure tubes, etc.

Graduated measuring apparatus not sub-divided.

	Capacity ml.	10	25	50	100	250
A	Tolerance ± ml. ..	0·015	0·03	0·04	0·06	0·10
B	Tolerance ± ml. ..	0·03	0·06	0·08	0·12	0·20

Detailed regulations as to construction of vessels of each type are given for both classes, and it may be noted that vessels intended for delivery (excepting pipettes) are not accepted for Class A; cylinders are not accepted for delivery for either Class A or B; and only flasks of over 100 ml. capacity for B.

Apparatus not specifically dealt with in the pamphlet may be submitted for testing and is subject to special terms.

Vessels graduated in "G.W.A." units, that is Mohrs grammes or cubic centimetres (1000 G.W.A. = 1002 ml.) are accepted for either class A or Class B tests, and the tolerances given above for vessels graduated in millilitres apply, and a special mark 𝔐A or 𝔐B is used.
'24. '24.

The pamphlet concludes with a list of verification and other charges.

D. G. H.

Joint Committee for the Standardisation of Scientific Glassware.

REPORT No. I. ON UNITS OF VOLUME.

THE Joint Committee for the Standardisation of Scientific Glassware, working under the aegis of the Institute of Chemistry, and having members representing the users, manufacturers, dealers, and Government Departments interested in scientific glassware, has issued the subjoined report:*

REPORT ON UNITS OF VOLUME.

The Committee have given much careful consideration to the question of units of volume, and as a result of their deliberations they unanimously recommend:

> *That the recognised international metric units—the "litre" (l) and "millilitre" or thousandth part of the litre (ml)—shall be used as the standard units of volume, and that standard volumetric glassware shall be graduated in terms of these units and marked "ml" instead of "c.c."*

In making this recommendation, the Committee have had in mind the great advantages to be gained by the general use of a uniform system which has been recommended by many scientific bodies, and is at present already extensively used, notably in America. The substitution of the millilitre for the cubic centimetre involves no practical difficulties as the two units differ only very slightly in magnitude. The difference in capacity between a 1000 c.c. and a 1000 ml. flask is, for example, only one seventh of the permissible error on a standard 1000 ml. flask.

In the course of the discussions, however, it became evident that some chemists still prefer to use Mohr's system of units, and would be unwilling at present to change their existing practice. It was clear, therefore, that some temporary inconvenience would be caused by an immediate complete withdrawal of official recognition of Mohr's units.

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* Copies of this Report may be obtained from the Secretary of the Committee, Institute of Chemistry, price 6d., post free.

In face of these facts the Committee have felt it desirable to couple with the above recommendation certain proposals designed to remove the confusion at present existing between the two systems of units. These are:

(1) That the unit of volume on Mohr's system shall in future be designated "G.W.A.,"[*] and that these letters shall be marked on all volumetric glass vessels graduated on the basis of Mohr's system, and shall be used by chemists when reporting results obtained by the use of such vessels.

(2) That the "G.W.A." unit shall be defined thus:

1000 G.W.A. = 1002 ml.

These additional recommendations will enable the two systems to be used side by side with a minimum of inconvenience during the transitional period which the Committee realise must precede the universal adoption of the litre and millilitre for general chemical work.

The Committee understand that in view of the above recommendations the National Physical Laboratory will accept for test[†] vessels graduated on the basis of G.W.A. units, and clearly marked to indicate this, and will place a distinctive mark on approved vessels graduated on this system, but not the Laboratory monogram NP, which will be reserved for vessels graduated in millilitres.

Information relating to the two systems of units, and the reasons underlying the above recommendations are given in the following notes.

METRIC UNITS OF VOLUME.

The volume of a body of simple geometrical shape can readily be calculated from the linear dimensions of the body. Hence, once the unit of length has been decided upon, the unit of volume should theoretically be defined in terms of the unit of length. The volume of a cube each of whose sides is of unit length becomes automatically the unit of volume; such a definition by its logical simplicity makes an instant appeal to the mind, and it is therefore natural to find in common use such units as the cubic centimetre and the cubic foot, based on the corresponding units of length.

Volumetric measurements, however, are most extensively used when dealing with fluids, and hollow vessels of known capacity have long been used for measuring volumes of liquid. But while it is extremely difficult to determine accurately the capacity of a hollow vessel from measurements of its internal dimensions, to determine the weight of liquid required to fill a vessel is, on the contrary, an operation which may be carried out easily and to a high degree of precision. Hence, units of volume have come into use which are not defined in terms of the units of length, but as the volumes occupied by specified weights of a particular liquid—usually water. The gallon and the litre are units of this kind.

Two distinct types of units of volume have thus come into general use. Sooner or later under any system the necessity arises of determining the relation between the two types of units, of determining for example, the number of gallons in a cubic foot. In the older systems of units the two types of units of volume were defined independently of each other, and as a result no simple numerical relation exists between them. The number of gallons in a cubic foot, for example, is not represented by a simple whole number.

One of the objects of the founders of the Metric System was to secure a simple numerical relation between the two types of units of volume. It was intended to achieve this by defining the unit of mass, the kilogramme, as the mass of a quantity of water which at its temperature of maximum density occupied a cubic decimetre. The unit of volume, the litre, could then be defined indifferently either as a cubic decimetre or as the space occupied by a kilogram of water at its temperature of maximum density.

A cubic decimetre of water is, however, manifestly unsuitable for practical use as a standard weight. To give practical effect to the system it was necessary to make a standard kilogramme weight whose mass should be in accordance with the definition laid down. A standard kilogramme weight—the "Kilogramme des Archives"—was prepared with great care and skill at the end of the eighteenth century, but during the nineteenth century distrust in the accuracy of the work arose. Consequently in 1872 the *Commision Internationale du Metre* discussed the matter fully in all its bearings and decided that the International Kilogramme should be deduced from the Kilogramme des Archives in its actual state. The original definition of the kilogramme was thus abandoned, and the kilogramme was re-defined as the mass of a particular standard weight.

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[*] Vide *infra.*

[†] A pamphlet giving details of the tests on volumetric glassware carried out at the National Physical Laboratory may be obtained free of charge on application to The Director, The National Physical Laboratory, Teddington, Middlesex.

The definition of the litre was subsequently revised* in conformity with this change, and the accepted definition at the present time is:—

"The unit volume for determinations of high precision is the volume occupied by a mass of one kilogramme of pure water at its temperature of maximum density and under normal atmospheric pressure†; this volume is termed the litre."

It is important to observe that the definition of the litre is now absolutely independent of the metric units of length. The cubic centimetre, on the other hand, being simply the volume of a cube whose edges are one centimetre long, is completely defined without any reference to the volume of a mass of water. Thus no direct relation between the litre and the cubic centimetre results from the definitions of these units. This relation can only be obtained by direct experimental determination, and the most accurate determination available is that carried out at the Bureau International des Poids et Mesures. M. Benoit‡ in his summary of the work gives as the most probable value

$$1 \text{ litre} = 1000{\cdot}027 \text{ c.c.}$$

and states that the uncertainty of this value probably does not exceed one unit in the last decimal place.

The difference between the cubic centimetre and the millilitre is so small as to be negligible for ordinary volumetric glassware; the difference in capacity between a 1000 c.c. flask and a litre flask, for example, is less than the observational errors liable to arise in the ordinary use of either flask.

The term "cubic centimetre" has, however, been misapplied by the users of Mohr's system (see below) to denote the volume of a quantity of water having an apparent weight in air of 1 gramme. A flask which contains an amount of water weighing 1000 grammes in air has a capacity of almost exactly 1002 c.c. To speak of such a flask as a 1000 c.c. flask is clearly inadmissible. Volumetric glassware, manufactured by the leading British firms, and marked "c.c.," may safely be relied upon as having been calibrated on the basis of the cubic centimetre. Volumetric glassware of foreign manufacture similarly marked "c.c.," may have been calibrated in terms of either the cubic centimetre or grammes of water.

In view of this confusion in the use of the term cubic centimetre, and of the fact the cubic centimetre is not exactly one thousandth part of a litre and further, because the mass of water which occupies 1 c.c. under definitely specified conditions cannot be directly derived from the accepted definition of the litre, the Committee adopted the recommendation given above, *viz.*:—

> That the recognised international metric units—the *litre* (l) and *millilitre* or thousandth part of the litre (ml)—shall be used as the standard units of volume, and that standard volumetric glassware shall be graduated in terms of these units and marked *ml.* instead of *c.c.*

The acceptance of this recommendation by chemists and manufacturers of volumetric glassware would bring the practice in this country into line with that in other countries, notably America, and would give effect to the recommendation of numerous scientific bodies that the litre, as defined above, should be exclusively used as the unit of volume in all scientific work. The publication of all results in terms of millilitres would secure a uniformity which is at present lacking, and remove the uncertainty due to the term cubic centimetre being used sometimes correctly, sometimes as equivalent to the millilitre, and sometimes as the space occupied, under conditions not generally well defined, by a quantity of water which has an apparent weight in air of one gramme.

MOHR'S SYSTEM.

According to Mohr's original proposal a "litre" flask would be adjusted so that when filled with water at 17·5° C. it would contain an amount of water which, when weighed in air against brass weights, had an apparent weight of 1000 grammes. Such a flask would contain almost exactly 1002 c.c. The term cubic centimetre was misapplied to the thousandth part of this volume. This misuse of both the terms litre and cubic centimetre still persists.

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* Trav. et Mem., Vol. 12, 1920.

† The reference to normal atmospheric pressure perhaps requires a word of explanation. Water is slightly compressible, and hence the volume of any given quantity of water varies slightly with the pressure to which it is subjected. Hence, to be precise, the definition of the litre must state the pressure to which the water is to be subjected.

‡ Trav. et Mem., Vol. 14, 1910.

Some chemists still maintain that it is more convenient in practice to use as a unit of volume the space occupied by a quantity of water which has an apparent weight of 1 gramme in air at ordinary room temperature.

To avoid confusion resulting from the use of the terms "litre" and "cubic centimetre" in connection with such units, the Commtitee recommend (see (1), page 2) that the letters "G.W.A." —a mnemonical abbreviation for "grammes of water in air"—shall be used in describing such units.

It is further necessary that an exact definition of the "G.W.A." unit be adopted.

A quantity of water which weighs 1000 grammes in air of density 0·0012 grms./ml. when weighed against brass weights of density 8·4 grms./ml. occupies a volume of 1002·021 ml. at 60° F. This volume differs from 1002 ml. by only two parts in a hundred thousand, which is negligibly small in comparison with the tolerances allowed on volumetric glassware, and the accuracy attainable in the ordinary use of such glassware. Hence the committee on grounds both of convenience and precision have adopted the definition given in recommendation (2), above, namely:

$$1000 \text{ G.W.A.} = 1002 \text{ ml.}$$

This relation defines the G.W.A. unit in a simple and accurate manner, and at the same time gives it a value which differs only by a negligible amount from the volume occupied at room temperature (60° F.) by a quantity of water which has an apparent weight of 1 gramme when weighed in air against brass weights.

The above definition enables conversion from millilitres to G.W.A. units and vice versa to be easily made, and will enable the two units to be used side by side without confusion during the transitional period which must elapse before the universal adoption of the litre and millilitre is obtained in this country.

August, 1924.

Carbon Monoxide in Public Gas Supplies

REPORT TO THE BOARD OF TRADE.*

FROM 1812, when a public gas supply was started in London, until about 1840, the gas was produced by the carbonisation of coal in iron retorts, and contained from 3 to 8 per cent. of carbon monoxide. By about 1860 fireclay retorts had been generally adopted, and the proportion of carbon monoxide consequently increased by about 1 to 2 per cent. The average proportion throughout the country from about 1860 to the early nineties may be taken to have been 7 to 8 per cent., and nearly 30 per cent. of the gas still sold in England and Wales is such coal gas without admixture. If a gas undertaking makes only ordinary coal gas it has to find a market for a relatively large quantity of coke (75 to 80 lbs. for each 1000 cb. feet of gas it sells). Hence, if the coke does not return a fair proportion of the cost of the coal, more must be added to the price of the gas. The alternative is to use some of the coke for gas-making, and the gas made from coke, by any process practically available, is of the general character of water-gas and contains considerably more carbon monoxide than ordinary coal gas.

In 1921 the quantity of water-gas made by authorised gas undertakings in England and Wales was 48,130 million cb. feet, in the production of which 1,207,000 tons of coke and 51,573,577 gallons of oil were used. To obtain coal gas equivalent in heating power to this water gas about 3 million tons of coal would have had to be added to the 14,168,094 tons carbonised in 1921, and a market would have had to be found for an additional 2,700,000 tons of coke.

Computation of the Proportion of Carbon Monoxide in Gas. Adopting the average amount of 8 per cent. for the carbon monoxide in coal gas, and 42 per cent. for the carbon monoxide, in uncarburetted or blue water gas (which percentage, when the gas is carburetted, is diminished by about 4 for every gallon of oil used per 1000 cb. feet of the carburetted gas), it is possible to calculate the average proportion of carbon monoxide in the gas made by any undertaking over a given period, by the use of the formula—

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* By W. J. A. Butterfield (one of the Gas Referees). With correspondence arising thereon. H.M. Stationery Office. 1924. 2s. 6d. net.

$$\frac{8C+(42-4X)W}{100}$$

in which C is the percentage of coal gas, W the percentage of water gas (blue or carburetted), and X the number of gallons of oil used per 1000 cb. feet of carburetted water gas made. Since the mixed gas consists only of coal gas and water-gas (blue or carburetted), $W=(100-C)$.

This formula gives results sufficiently accurate for all necessary deductions as to the toxicity of public gas supplies in England and Wales at different times. It is not, without modification, applicable to Scotch supplies, because the coal gas, as produced from most Scotch coals, contains more than 8 per cent. of carbon monoxide.

Spasmodic analyses of the gas supplied in any area give little information as to the average proportion of carbon monoxide over a period of, say, 12 months. All that is needed for the application of the formula, however, is that gas undertakings should be under obligation to supply to the Board of Trade certified statements of the quantities of coal gas and water-gas made, and of coal and oil used in the manufacture, during the period of enquiry. Any gas obtained per ton of coal, in excess of 12,700 cb. feet, may be regarded as water gas in so far as its content of carbon monoxide is concerned, without risk of any serious error being incurred. This formula has been utilised for an exhaustive investigation into the proportions of carbon monoxide in the gas supplied by practically every gas undertaking in England and Wales during the past ten years.

Fatalities from Gas Poisoning.—Definite information has also been obtained as to the number of gas poisoning fatalities from the Registrar-General's Department. From a consideration of the various factors involved the following conclusions (*inter alia*) have been drawn:—The death rate from accidental gas-poisoning in the last 10 years is approximately one per annum per 350,000 to 400,000 persons. It is much lower in the small towns and villages, and this difference appears to be determined, not by the proportion of carbon monoxide, but by differences in housing conditions, uses to which the gas is put, and condition of gas pipes and fittings. Prior to the last five or six years the steady increase in the proportion of carbon monoxide in the gas was not attended by any increase in the death rate from gas poisoning, whilst the slight increase in the death rate during the last five or six years (during which there has been an increase in the proportion of carbon monoxide in the gas supplied) coincides with the advent of a period of neglect of internal gas piping, fittings, etc., in the case of supplies through ordinary meters. On the other hand, an extremely low death rate is associated, with prepayment meters, for the pipes and fittings of which the gas undertakings are usually responsible.

RECOMMENDATIONS.—The following recommendations are made, as following from the conclusions drawn in this Report:

I. That the inquiries instituted as from January, 1922, by the Board of Trade, in collaboration with the Ministry of Health, into the circumstances of every fatality from poisoning by gas from public supplies, be continued; the information afforded thereby be incorporated in periodical reports, and be utilised as a guide in formulating measures for reducing the risks of gas poisoning. The reports might suitably cover periods of three or five years.

II. That the gas industry be asked to submit to the Board of Trade at an early date suggestions for a system of periodical inspection, and of maintenance in safe condition, of gas piping, fittings and appliances in dwelling houses.

III. That the suggestions received accordingly, subject to such additions or modifications as the Board of Trade may think necessary, be the basis of a scheme of inspection and maintenance of gas piping, fittings and appliances, which all gas undertakings specified by the Board of Trade shall be required to adopt and carry into effect without delay.

IV. That the gas industry be asked, after conferring with the makers of gas fittings and appliances, to submit to the Board of Trade proposals for the elimination from the trade of (1) types and qualities of fittings, etc., which are liable, after a few years' use, to become unsound or otherwise unsafe; and (2) flueless "heaters" or other appliances which are apt to evolve carbon monoxide. Also to submit proposals for preventing geysers, etc., being fitted in very small rooms without adequate and trustworthy vents to the open air for the products of combustion.

V. That these proposals, subject to such additions or modifications as the Board of Trade may think necessary, be recommended for adoption forthwith by all gas undertakings, and by all makers, dealers in and vendors of gas fittings and appliances in this country. That if such recommendation fails to prevent, within one year or at most two years, the supply to gas consumers of untrustworthy or unsafe gas fittings and appliances, the Board of Trade shall take steps (in regard to which it may ask, if necessary, the assistance of an advisory Committee) to secure to Local Authorities powers to stop the sale and supply in their districts of gas fittings and appliances which do not fulfil such requirements as to trustworthiness and safety as may be laid down from time to time in such manner as it may direct.

Ministry of Agriculture and Fisheries.

INVESTIGATIONS INTO THE CAUSES OF THE UNUSUAL MORTALITY IN ENGLISH OYSTER BEDS IN 1920 AND 1921.*

The possible causes investigated include (1) direct poisoning, (2) disease due to parasites, (3) weakening of the oysters due to the abnormal summer, with subsequent natural death, (4) unusual attack by marine organisms. No definite cause has been traced; no single organism has been found common to all the dying oysters, nor any single symptom, neither has any evidence been found indicating that death might be due to T.N.T. poisoning resulting from the dumping of explosives, although it is possible that the enquiry was started at too late a stage for the detection of this.

The following test has been devised to detect 1 part of T.N.T. in 50 millions when two litres are available. The water is acidified with 25 c.c. of 10 per cent. sulphuric acid and extracted with 50 c.c. of benzene; after separation the benzene is evaporated to 5 c.c., 20 c.c. of alcohol are added and then 2 c.c. of 10 per cent.

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* Part II. Chemical Reports, etc. (Ministry of Agriculture and Fisheries, Ser. II., Vol. 6, No. 4, 1924). Pp. 69. H.M. Stationery Office. Price 7s. 6d. net.

sodium hydroxide solution, when the development of a fugitive red or pink colour indicates the presence of T.N.T. The colour so obtained gradually changes to brown, and is available after half an hour for quantitative estimation by matching against standard solutions of T.N.T. similarly treated.

Bacteriological tests on healthy specimens indicated total organisms growing on agar at 20° C. from 250 to 8000, and generally the presence of *B. coli* in fair numbers; cladothrix and streptococci are usually absent. Arsenic is present in oysters, especially in those from certain beds, those from Whitstable containing in some cases as much as 3·7 parts per million, and a few contain copper and zinc. Numerous detailed chemical and bacteriological analyses are given. The following is a typical analysis of oyster meat: Water, 78·7; fat, 1·9; proteins, 11·2; carbohydrates, 6·0; ash, 2·2; glycogen, 4·4 per cent.

H. E. C.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Estimation of Maltose in Presence of other Reducing Sugars by means of Barfoed's Solution. P. Nottin. (*Comptes rend.*, 1924, **179**, 410–413.)—Contrary to Le Grand's statement that maltose does not reduce Barfoed's solution under conditions specified (ANALYST, 1921, **46**, 406), the author finds that the reduction effected by maltose in less than 1 per cent. solution is equal to that caused by 7 per cent. of its weight of dextrose, and diminishes as the concentration of the maltose increases. On the average the reduction of Bertrand's solution by maltose is equal to that of 57·2 per cent. of its weight of dextrose; this ratio varies with the concentration of the maltose, but may be calculated from Bertrand's tables. Thus the proportions of maltose and dextrose present in a solution containing about 0·5 per cent. of reducing sugars, expressed as dextrose, may be calculated as follows:—If A and T are the reducing powers of the solution determined according to the exact methods given by Le Grand and Bertrand respectively, and G and M the amounts of dextrose and maltose, $A = G + 0{\cdot}07M$ and $T = G + 0{\cdot}572M$, from which $M = 1{\cdot}995(T - A)$ and $G = A - 0{\cdot}07M$. Application of this procedure to various mixtures of dextrose, maltose and dextrin has given results in accord with polarimetric measurements.

T. H. P.

Chocolate in Chocolate-Coated Tablets and Pills. E. S. Rose. (*Amer. J. Pharm.*, 1924, **96**, 590–592.)—Chocolate was detected by removing the coating from 3 to 10 tablets with warm water, slightly acidifying with acetic acid, boiling, adding 1 grm. of precipitated chalk, filtering, and washing with warm water. Five c.c. of 10 per cent. sodium carbonate solution were then added to the precipitate, which was boiled for 2 minutes, filtered off, and washed with 2 c.c. of water, the filtrate acidified with acetic acid, treated with 3 c.c. of copper sulphate solution (5 grms. copper sulphate, 1 c.c. glacial acetic acid, and 100 c.c. of water) allowed to stand half-an-hour and centrifuged for 2 to 3 minutes. A brown flocculent precipitate indicates cocoa, and 5 mgrms. of cocoa may thus be detected if the mixture is allowed to stand several hours before centrifuging. Ferric oxide, burnt umber, extract of liquorice, coal-tar colours, chalk, cane sugar, glucose, gelatin, gum arabic, starch, etc., do not interfere. It was found that cocoa was present in less than half the brown-coated samples examined, the amount being probably less than 5 per cent. of the coating used. D. G. H.

Microchemical Detection of Acetaldehyde in Fruits. C. Griebel. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 438–441.)—Acetaldehyde, which has been shown to be present in apples and stone fruits, may be detected even in minute quantities microchemically by means of the characteristic crystals which it forms with *p*-nitrophenylhydrazine. A tiny fragment or section of the fruit is placed in a small cell formed by a piece of barometer tubing about 15 mm. long and a micro-slide; to it are added a minute crystal of the reagent and a drop of dilute acetic acid (15 per cent.), and the whole is very gently warmed with a cover slip over the top for about 30 seconds. Characteristic crystals of acetaldehyde-*p*-nitrophenylhydrazine are sublimed on to the cover slip, which may be examined under 100 magnifications. The crystals are small yellow needle-like prisms, frequently sabre-shaped. The test, as so applied, will detect as little as 0·002 mgrm. of acetaldehyde or, say, 0·05 per cent. in a fragment of fruit. Certain other aldehydes or ketones, such as acetone, benzaldehyde or furfuraldehyde, give a similar reaction, but the colour and shape of the crystals are different.
H. E. C.

The Solanine Content of the 1922 Crop of Potatoes. C. Griebel. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **47**, 436–438.)—A serious outbreak of potato poisoning occurred in Germany in the autumn of 1922 which was traced to an abnormal amount of solanine in the potatoes raised in that year (*cf.* ANALYST, 1923, **48**, 454). Some specimens contained as much as 0·79 per cent., as compared with a content of about 0·05 per cent. in the 1923 crop; this is attributed to the abnormally wet weather then prevailing, and, secondly, to the influence of light during storage or exposure for sale. Those tubers or parts exposed to the light contained much more solanine than other parts, so that, of the same potato, one part may be poisonous and another not so. (*Cf.* also ANALYST, 1924, 284.)
H. E. C.

Molecular Combination of Caffeine and Salicylic Acid. A. Regenbogen and N. Schoorl. (*Pharm. Weekblad*, 1924, **61**, 34–36.)—Equimolecular weights of caffeine and salicylic acid, when fused together, form a compound termed caffeine-salicylic Acid. It can be crystallised from water or chloroform in the form of microscopic, dextro-rotatory crystals, m.pt. 137°. Evidence points to the fact that "Salicylas Natricus cum Caffeine" of the Netherlands Pharmacopoeia, known as caffeine-sodium salicylate, is in reality the sodium salt of the caffeine-salicylic acid obtained by the author. W. S. S.

Reaction of Antipyrine with Trichloracetic Acid. L. Debucquet. (*J. Pharm. Chim.*, 1924, **30**, 121–122.)—Antipyrine and trichloracetic acid are dissolved in weights proportional to their molecular weights in equal volumes of water, the solutions mixed and vigorously shaken, and the resulting viscous mass and supernatant liquid allowed to crystallise for 2 or 3 days, when an opaque crystalline block will have formed. Re-crystallisation, with slow cooling, results in the formation of anhydrous shining plates melting at 79–80° C., easily soluble in alcohol, chloroform, ether and benzene; slightly soluble in petroleum spirit; giving acid solutions showing all the colour reactions of antipyrine with ferric chloride and sodium nitrite, and negative results with acidified silver nitrate solution. Owing to the instability of the solution the trichloracetic acid may be estimated directly with 0·1 *N* sodium hydroxide solution, and the antipyrine by Bougault's volumetric method, after eliminating the trichloracetic acid. Results obtained agreed with the formula $CCl_3COOHC_{11}H_{12}N_2O$ and a molecular weight of 351·5.

D. G. H.

Commercial Anhydrous Quassine. Rengniez. (*J. Pharm. Chim.*, 1924, **30**, 120–121.)—Since the first process in the preparation of crystalline quassine produces a variable extractive termed "amorphous quassine," but differing entirely in properties from either crystalline, or the true amorphous quassine produced from the crystalline variety, and containing from rather less than one-tenth to over two-tenths of crystalline quassine, the author advocates the exclusion of such a varying product from the French Pharmocopoeia. D. G. H.

Detection of Diethyl Phthalate. H. Leffmann. (*Amer. J. Pharm.*, 1924, **96**, 503–505.)—Diethyl phthalate may be used as a denaturant for commercial alcohol, and a comparison of the methods used in its detection have shown that good results are obtained by Lyon's modification of Utz's method, in which 1 c.c. of the sample and 1 c.c. of concentrated sulphuric acid are heated together till the alcohol is driven off, slightly cooled, a pinch of resorcinol added and the mixture heated till it becomes reddish-brown, after which it is cooled, excess of ammonium hydroxide solution cautiously added, and the whole poured into 500 c.c. of water, when fluorescence will result if the phthalate was present. Andrews' method, however, (ANALYST, 1923, **48**, 508) is particularly simple and dependable. Acetaldehyde, commercial methyl alcohol and commercial isopropyl alcohol in quantities likely to be present in recovered or imperfectly prepared alcohol were not found to interfere in Andrews' method. D. G. H.

Biochemical, Bacteriological, etc.

Haemolysis, as a means of Detecting the Adulteration of Milk. M. J. N. Schuursma. (*Chem. Weekblad*, 1924, 365–367.)—Haemolysis has been found to take place in milk of a certain dilution, and a method, known as the "Erythrocyte method" was devised by the author for the detection of dilution of milk. It has an accuracy comparable with that of the usual method, and at the same time, requires a shorter time for the estimation. The method is as follows:—Preliminary experiments are carried out on a normal (undiluted) milk. Portions, each of 10 c.c., of the normal milk are pipetted into a number of dry test tubes, and distilled water, varying in amount from 1 c.c. upwards, added; 5 drops of fresh ox blood are added, the solution well mixed, and, after standing for 15 minutes, centrifuged for ar least 20 minutes. The dilution at which haemolysis begins is indicated by the first appearance of a red coloration in the supernatant liquid. The experiments are repeated, varying the dilution of the milk to approximate to the dilution already found to produce haemolysis, and thus an accurate estimation can be made. The milk to be tested, is now diluted in exactly the same manner, and the point at which haemolysis begins should correspond to that already determined in the blank. Dilution of the milk may be detected by the increase of haemolysis as compared with the blank. W. S. S.

Application of Permutit to the Separation and Estimation of Ammonia in Urine. A. Kolb. (*Chem. Zeit.*, 1924, **48**, 557.)—Folin and Bell (*J. Biol. Chem.*, 1917, 29, No. 2) published a paper on the colorimetric estimation of ammonia in urine with synthetic zeolite (permutit). Permutit as prepared by Gans (*Chem. Zeit.*, 1907, 355; Kolb, *Chem. Zeit.*, 1911, 1393, 1410, 1419), after being subjected to special treatment, has been changed into a pure, moderately fine, insoluble powder, which, in contact with water, gives no dusty constituents, and settles quickly after being shaken with water. Basic ammonia contained in a liquid may be removed easily with permutit, and other organic, nitrogenous materials contained in urine are removed by being washed out from the permutit. The taking up of ammonia by permutit is an exchange of ions and not a case of absorption; through the action of another salt solution, *e.g.*, sodium chloride solution, the base can be made free again. The new colorimetric method is described in detail. It is simple and quickly and easily carried out. After the ammonia has been freed by the addition of sodium hydroxide solution it is estimated colorimetrically in the usual way with Nessler's reagent. A large number of comparative estimations of ammonia in pathological urine gave corresponding values by the new colorimetric and the distillation methods, but the former was always more rapid. P. H. P.

Efficiency of some Organic Dyes as Anti-ferments. E. H. Harvey. (*Amer. J. Pharm.*, 1924, **96**, 585–589.)—Since yeast gradually hydrolyses dextrorotatory sucrose to equal parts of dextrose and laevulose, the reaction may be followed by the change in hourly polarimetric readings (ANALYST, 1923, **48**, 131, 232), and the efficiency of anti-ferments will be inversely as the increase in the difference between readings. Of the dyes tested, safranine was found to be the most efficient anti-ferment, followed by paranitrophenol, methylene blue, eosin Y, aniline violet (methyl), cyanine and fuchsin, whilst alizarine blue had no anti-ferment effect. A table is given showing the relative efficiency on a percentage basis of the various anti-ferments tested on this and previous occasions, together with an extensive bibliography on the use of dyes as bactericides. D. G. H.

Detection of Koch's Bacillus in Ordinary Media. G. Pégurier. (*Ann. Chim. anal.*, 1924, **6**, 226–229.)—A fragment of sputum is warmed on a slide with carbol-fuchsine solution, freed from excess of the stain, treated with an aqueous alcoholic solution of lactic and picric acids, washed with water, and with 95 per cent. alcohol until the yellow colour of the picric acid appears, and then counter-stained with Esbach's aqueous citro-picric acid reagent. After removal of the excess of the stain, the Koch's bacilli, coloured scarlet, stand out clearly from the other bacteria and tissue elements, which appear saffron yellow. In most instances this procedure indicates a greater number of Koch's bacilli than are rendered evident by the Ziehl-Nielsen method. T. H. P.

Fermentation of Pentoses by *Bacillus Granulobacter Pectinovorum*. W. H. Peterson, E. B. Fred and E. G. Schmidt. (*J. Biol. Chem.*, 1924, **60**, 627–631.) —Speakman (*J. Biol. Chem.*, 1923–24, **58**, 395) has classified the carbohydrates into two groups, those which ferment normally and those which do not. Glucose and starch illustrate normal fermentation, in which the acidity reaches a maximum, falls rapidly, then slowly rises again. Xylose and arabinose he placed in the group producing abnormal fermentation; high acidities with little or no break in the curve were found with both pentoses. No quantitative data on solvent production are given by him. The authors find that the fermentation of xylose and arabinose by *Bacillus granulobacter pectinovorum* results in the same products and in essentially the same quantities as from glucose. Slightly less solvents and slightly more volatile acids are produced from the pentoses. The rate of fermentation is somewhat slower for the pentoses than for glucose; but practically all the sugar is destroyed in 72 hours. The destruction of sugar and production of solvents do not agree with Speakman's statement that abnormal fermentations (xylose, mannitol, etc.) are characterised by a low consumption of carbohydrates and a poor yield of solvents. P. H. P.

Determination of the Number of Bacteria in Soil. C. L. Whittles. (*J. Agric. Sci.*, 1924, **14**, 346–369.)—The author has shown in a previous paper (ANALYST, 1923, **48**, 280) that the frequency of vibration has a profound influence on the disintegration of soil particles, and hence on the bacteriological count as obtained by plating out in the usual way. A new apparatus is now described by which the frequency may be varied at will and measured. The degree of disintegration following vibration, with and without shaking and trituration, has been measured by a modification of Robinson's depth concentration method (ANALYST, 1923, **48**, 83). Vibration alone is not satisfactory, but should be preceded by trituration with a rubber-covered pestle. The rate of wetting is an important factor; the use of acids is not advisable, but ammonia may be used.

H. E. C.

Toxicological and Forensic.

Distribution of Lead in the Living Organism. I. A. Christiansen, G. Hevesy and S. Lomholt. (*Compt. rend.*, 1924, **179**, 291–293.)—The determination of the distribution of lead in different organs of the body has been made by the radio-activity method already described for bismuth (*Biochem. J.*, 1923, **17**, 441). It consists in administering lead containing a known amount of radium D, then estimating the lead in the different organs by decomposition with nitric acid, and measuring the equilibrium between the lead contained therein and the β-rays of radium E by means of an electroscope. The results show that the greater part of the lead accumulates in the liver, whence it is slowly eliminated in the fæces; considerable amounts are also found in the intestines and kidneys.

H. E. C.

Solubility of Various Lead Compounds in Blood Serum. L. T. Fairhall. (*J. Biol. Chem.*, 1924, **60**, 481–484.)—It is of fundamental interest to know how lead is absorbed from the mucous membranes and respiratory tract and transported in the circulation. This investigation was carried out to determine whether the solubility of lead compounds in the fluids of the tissues is different from that in pure water. Blood serum (horse serum) was used as a representative medium, and in it the solubilities of metallic lead and certain lead salts were determined. The solubilities were also determined in serum saturated with carbon dioxide. Experimental details and tables of results are given. No marked increase in solubility was noted with the serum except in the case of lead oxide. Whereas in water its solubility is 17 mgrms., in serum it is 1·15 grms. per litre. Lead oxide and lead fume, both being readily soluble in serum, would be expected to find an easy mode of entrance into the circulatory system by forming lead hydroxide and combining with protein to form an alkali metaprotein. The proportion of those suffering from lead encephalopathy is higher in the manufacture of lead oxides than in any other lead industry. Possibly the ready solubility of these compounds allows lead to enter the system more quickly than it can be eliminated or deposited in the bones as relatively harmless lead phosphate, and the large quantities circulating tend to produce severe lead poisoning. P. H. P.

Rapid Method of Testing Urine for Lead. L. T. Fairhall. (*J. Biol. Chem.*, 1924, **60**, 485–488.)—Obvious methods for the analysis of urine and the estimation of its lead content are discussed and shown to be unsatisfactory. The author has devised a method for the quantitative precipitation of lead from urine by mechanical precipitation with the earthy phosphates. If fresh or well preserved urine is made strongly ammoniacal, and the mixture left overnight at room temperature in a large Erlenmeyer flask, the alkaline earth phosphates precipitate in a gelatinous mass so completely that the clear liquid above can be decanted. The precipitate can then be rapidly separated by suction filtration, collected, ashed and the amount of lead present then estimated volumetrically with greater facility than by the usual method of evaporating the urine to dryness and ashing the residue. Known amounts of lead were added to urine, in a series of experiments, before the addition of ammonia, and the recoveries were satisfactory. The method was applied to the urine of patients suffering from lead-poisoning, and results given show that the lead is all precipitated and the filtrate is lead-free. The urine must either be freshly collected or well preserved with powdered thymol, and heating should be avoided. P. H. P.

Water Analysis.

Colorimetric Estimation of Lead and Copper in Water. C. Pyriki. (*Zeitsch. anal. Chem.*, **1924, 64,** 325–330.)—Winkler's sulphide method (*Zeitsch. angew. Chem.*, **1913, 26,** 38) for the estimation of lead and copper gave good results for lead up to 0·002 grm. per litre; the results for copper should be multiplied by 0·813, as copper sulphide has a stronger colouring power than lead sulphide. With larger quantities of lead the results were much too low; this was proved to be due to the cyanide added, which prevents quantitative conversion into lead sulphide, probably by the formation of lead cyanide; the interference is not removed by addition of the sulphide first and the cyanide afterwards. The method was quite satisfactory in absence of copper, no cyanide being required. Winkler's ferrocyanide and sulphide method (*loc. cit.*) for the simultaneous estimation of copper and lead was found to be trustworthy. W. R. S.

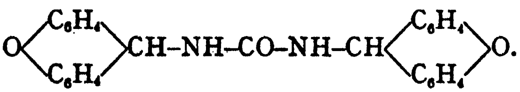
Agricultural Analysis.

New Method for the Estimation of Cyanamide in its Calcium Compound. R. Fosse, P. Hagene and R. Dubois. (*Comptes rend.*, **1924, 179,** 408–410.)—When heated for 3 hours at 50° to 55° C. with 2*N*- or *N*-nitric acid, silver cyanamide yields quantitatively the corresponding amount of urea, the estimation of which then gives the cyanamide to within 0·5 per cent. With calcium cyanamide, 1 grm. of the material is mixed with 10 c.c. of water and 50 c.c. of 2*N*-nitric (or hydrochloric) acid in a conical beaker, which is immersed for 3 hours in a bath at 50–55° C. The liquid is then cooled, rendered slightly alkaline with ammonia, filtered, washed into a 500 c.c. flask and made up to volume with water. Of this solution 20 c.c. are treated with 40 c.c. of acetic acid and 3 c.c. of a 10 per cent. solution of xanthydrol in methyl alcohol for 3 hours, the xanthylurea being then collected, dried and weighed. The results thus obtained differ but slightly from those furnished by Caro's method.

T. H. P.

Colorimetric Estimation of Humic Matter in Mineral Soils. T. Eden. (*J. Agric. Sci.*, **1924, 14,** 469–472.)—The method, which is a modification of that of Oden, estimates the total coloured organic matter of the soil, and includes the so-called humic acid and humin. Five grms. of the soil are treated in a Gooch crucible with 50 c.c. of 10 per cent. hydrochloric acid and well washed. The soil is then transferred, with the aid of about 60 c.c. of water, to a conical flask marked at 100 c.c., plus the volume of the soil, 20 c.c. of 50 per cent. sodium hydroxide solution are added, and the flask is filled to the mark (a few drops of alcohol may be used to clear the meniscus if necessary). It is then immersed in boiling water for 15 minutes and its contents constantly stirred. A portion of the hot solution is filtered through a hardened paper, and 10 c.c. of the filtrate are diluted to 200 c.c. and compared in a colorimeter with a standard solution of Merck's *acidum huminicum*, which may contain about 0·3 grm. of the dry material dissolved in a slight excess of sodium hydroxide solution and made up to 100 c.c. H. E. C.

Gravimetric Estimation of Cyanamide in the Form of Xanthyl-Urea. R. Fosse, P. Hagene and R. Dubois. (*Comptes rend.*, 1924, **179**, 214–216.)—Pure silver cyanamide may be estimated easily and accurately in the form of dixanthyl-urea:—



$$O\left\langle\begin{matrix}C_6H_4\\C_6H_4\end{matrix}\right\rangle CH\text{–}NH\text{–}CO\text{–}NH\text{–}CH\left\langle\begin{matrix}C_6H_4\\C_6H_4\end{matrix}\right\rangle O.$$

Silver cyanamide in contact with nitric acid (40° Bé.), freed from nitrous vapours, is completely transformed into urea nitrate. The yield is theoretical if the mixture is left overnight at ordinary temperature or is kept for 1½ to 2 hours at 38° to 40° C. If left overnight, water is added next day to the crystalline mass thus formed in a stoppered bottle, the contents are made slightly alkaline with concentrated ammonium hydroxide, and treated with acetic acid and with a 10 per cent. solution of xanthydrol in methyl alcohol. After 2 hours of condensation the xanthyl-urea is washed with alcohol on a filter funnel, dried and weighed directly. Its weight, divided by 10, represents the corresponding amount of cyanamide, and by 7, the amount of urea formed. Two methods, varying slightly, carried out at ordinary temperature are described in detail, and two other similar ones carried out at 38° to 40° C. Results cited show the errors to be small. P. H. P.

Estimation of Uric Acid in Poultry Excreta. H. E. Woodman. (*J. Agric. Sci.*, 1924, **14**, 413–427.)—The well-known processes for the estimation of uric acid in urine are not applicable to its estimation in poultry excreta, and the piperidine extraction method is not altogether satisfactory. The new method proposed consists in the following stages: the removal of pigment, the decomposition of ammonium urate by hydrochloric acid, extraction of the uric acid by means of lithia, precipitation of the uric acid by ammonium chloride, decomposition with acid and titration with 0·05 *N* potassium permanganate solution. The method is applied as follows:—Eight to ten grms. of the mass are stirred with about 30 c.c. of alcohol, and the alcohol is poured off through a filter, this being repeated twice more with alcohol, then with ether. The extracted mass and the material on the filter paper are mixed with 35 c.c. of water, heated to boiling, after which 5 c.c. of hydrochloric acid are added, and the boiling is continued until the volume is reduced to about 20 c.c.; then the beaker is set aside in a cool place overnight. The acid liquor is poured through a filter, and the mass washed once with a little cold water. The material is now boiled with about 40 c.c. of water and 5 c.c. of 5 per cent. lithia solution. After settlement of the undissolved matter the solution is poured through a filter, and the residue is re-extracted with more dilute lithia solution. The filtrate and washings are evaporated to 100 c.c., then 30 grms. of ammonium chloride are stirred in and, next morning, the ammonium urate is filtered off and washed with ammonium chloride solution. The ammonium urate is dissolved in 50 c.c. of hot water and 5 c.c. of hydrochloric acid, from which, on cooling, the uric acid separates out; it may be collected on a Gooch filter, dried and weighed, or (preferably) dissolved in dilute lithia solution, acidified with sulphuric acid, and titrated hot with 0·05 *N* potassium permanganate solution. H. E. C.

Organic Analysis.

Effects of varying Hydrogen Ion Concentration on Tannin Analysis. W. R. Atkin. (*J. Soc. Leather Trades Chem.*, 1924, 8, 425.)—A summary is given of various papers on this subject with the author's comments. He points out that the variations in the tannin content of a natural quebracho extract (increasing from 61·6 per cent. to 72·6 per cent. over a range of P_H 1·86 to 8·0) are caused by a decrease in the insolubles, the sum of tannin and insoluble remaining almost constant. In the case of mangrove the addition of hydrochloric acid, although increasing greatly the insolubles, appeared to convert some non-tannin into tannin, but with chestnut and oak extracts the addition of acid decreased the tans and increased the non-tans. Comparing the P_H of natural quebracho extract before and after detanning, the author quotes the figures 4·56 and 4·27 respectively, whereas other workers find 4·60 and 5·57. The amounts of 0·1 *N* sodium hydroxide solution required to bring the tannin solution to $P_H = 7·0$, as found by titration, failed to do so in actual practice, and he ascribes this to oxidation by air and subsequent reduction by the hydrogen electrode. It is pointed out that catechol tannins give distinct variation in results with the colorimetric, hydrogen electrode and quinhydrone electrode methods, whilst the pyrogallol tannins yield concordant results. R. F. I.

Inorganic Analysis.

Benzidine as an Analytical Reagent and an Indicator for a definite Oxidation Potential. I. M. Kolthoff. (*Chem. Weekblad*, 1924, 21, 2–4.)—The colour change to bluish-violet undergone by benzidine acetate in the presence of oxidising agents, such as dichromates, etc., is made use of in the determination of a given oxidation potential. Particular attention is given to the oxidation potential of mixtures of ferricyanide and ferrocyanide. Ferricyanide was found to give the reaction readily, whereas attempts with ferrocyanide were negative. In the case of a mixture of the two it was found that the colour change, which is an indication of the oxidation potential, was dependent on the ratio of the two components of the mixture, but was independent of the absolute amounts present. Thus, when the ratio of ferricyanide to ferrocyanide was 9:1, a bluish-violet colour was obtained with benzidine acetate after one hour, but as the ratio decreased the intensity of the coloration diminished, till at 1: 9 no colour was obtained. W. S. S.

Antimony Trichloride in Volumetric Analysis. F. Feigl and O. Schummer. (*Zeitsch. anal. Chem.*, 1924, **64**, 249–255.)—Antimony trichloride solutions are not oxidised by atmospheric oxygen, hence their reducing action remains constant. In the following volumetric methods the antimony solution is added in excess, which is subsequently measured by Györy's bromate titration; standardisation is effected against bromate. *Hypochlorite.* The reaction proceeds as follows:—$NaOCl + SbCl_3 + 2HCl = NaCl + H_2O + SbCl_5$. A measured excess of antimony solution is added to the hypochlorite liquor; the mixture is treated with hydrochloric acid and well shaken to dissolve any basic antimony compounds. The reaction is instantaneous, and the bromate titration can be carried out at once. *Chloride of lime.* A thin paste, prepared by trituration, is diluted to a definite volume and an aliquot part treated as above. *Ferricyanide.* The reaction is $2K_3Fe(CN)_6 + SbCl_3 + 4HCl = H_4Fe(CN)_6 + K_4Fe(CN)_6 + SbCl_5 + 2KCl$. The measured excess of antimony solution is added to the ferricyanide solution, followed by treatment with strong hydrochloric acid and warming until the colour of the liquid changes from reddish-brown to nearly colourless. If the colour change is slow, more hydrochloric acid should be added. The liquid is then cooled, treated with excess of zinc sulphate solution free from iron, and titrated with bromate. *Ferrocyanide* can be estimated in the same manner after conversion into ferricyanide; the solution is acidified with sulphuric acid (1: 4) and oxidised with excess of permanganate; this is destroyed by careful addition of oxalic acid. Ferricyanide and ferrocyanide can be estimated in presence of each other by the above methods. W. R. S.

Titanous Salts as Reducing Agents. E. Knecht. (*J. Chem. Soc.*, 1924, **125**, 1537–1546.)—The quantitative reduction of nitric to nitrous acid in the presence of titanous chloride and an acceptor in the form of a primary aromatic amine can be conveniently utilised under certain conditions to diazotise the amine. Reduction of α-nitronaphthalene with titanous chloride (5 per cent. in excess of the 4 molecules required to produce the mono-chloro compound) in the presence of hydrochloric acid (24 molecules) will produce a 94 per cent. yield of crude 4-chloro-α-naphthylamine, of melting point 94° C. Methods for the volumetric estimation of tetramethyldiaminobenzhydrol, phenolphthalein, gallein, alizarin S, tartrazine, isatin, and glucosazone by means of titanous chloride are described, and also for the accurate estimation of phenylhydrazine in the products of acid hydrolysis of glucosazone. D. G. H.

Estimation of Phosgene. H. G. Reeves. (*J. Soc. Chem. Ind.*, **1924, 43,** 279T.)—Two methods are given: (1) By passing the gas into **13** *N* sodium hydroxide solution, which decomposes it with formation of sodium chloride and separation of sodium carbonate, which are then both estimated; (2) by leading the gas through a heated combustion tube containing asbestos impregnated with pure zinc dust. After cooling, the zinc is washed out of the tube with dilute nitric acid, and the chlorine estimated by Volhard's method. Free chlorine as an impurity is readily estimated either by passing the gas through potassium iodide solution and titrating the liberated iodine with thiosulphate solution, or by shaking the impure gas with mercury in a bulb immersed in a freezing mixture, the gain in weight of the mercury after gasification of the phosgene indicating the amount of chlorine present. Free hydrochloric acid is best estimated by passing the adulterated gas first of all through a cold U-tube containing glass wool coated with pure zinc dust. This combines with the hydrochloric acid, but not with the phosgene. The zinc chloride formed is estimated as before, and the phosgene by methods 1 or 2 above. Phosgene can be readily concentrated and kept for examination by passing it through nitrobenzene, which absorbs large volumes of the gas at ordinary temperatures and immediately gives it off again on heating the solution on a water bath till gas evolution ceases. Total removal of the gas is then effected by passing a current of dry air through the heated solution. R. F. I.

The Thiosulphate Precipitation of Copper. Drawe. (*Chem. Zeit.*, **1924, 48,** 593.)—After the precipitation of copper sulphide by means of thiosulphate, the filtrate, if required for the estimation of other metals, must be subjected to evaporation and oxidation for the purpose of destroying the excess of precipitant. These manipulations can be obviated by adding a slight excess of sodium carbonate, precipitating the metals by addition of sodium sulphide, and dissolving the sulphide precipitate in hydrochloric acid. W. R. S.

Oxidation of Arsenious Acid in Alkaline Solution by Permanganate. F. Feigl and F. Weiner. (*Zeitsch. anal. Chem.*, **1924, 64,** 302–322.)—A critical study was made of this reaction, which is stated to take place according to $3As_2O_3+4KMnO_4=3As_2O_5+2K_2O+4MnO_2$. The authors find that the equation is correct only in presence of a constant excess of permanganate; they condemn as unreliable the methods of Reichard for the estimation of permanganate (and of lead peroxide) by means of alkaline arsenite solution, it being found that the manganese peroxide formed, as well as atmospheric oxygen, contributes to the oxidation of the arsenite. Reichard's methods should be excised from the textbooks (*e.g.* Rüdisüle, Vol. V., **462**; Classen, *Theorie und Praxis der Massanalyse*, **411–412**). Brauner's statement (*Zeitsch. anal. Chem.*, **1916, 55,** 225) that manganese sesquioxide is the product of the reaction could not be confirmed; the precipitate was found to be richer in oxygen than Mn_2O_3. W. R. S.

Use of Molybdenum as Indicator in the Volumetric Estimation of Zinc. L. Fernandes. (*Giorn. Chim. Ind. Appl.*, 1924, **6**, 334–335.)—Since, in acetic acid solution, alkali molybdates are not precipitated by potassium ferrocyanide, but give an intense red coloration, they serve as indicators in the estimation of zinc by titration with ferrocyanide, rendering the end-point more distinct, and thus diminishing the comparatively large error to which this method is subject. The zinc solution is evaporated to about 25 c.c., neutralised with ammonia, if containing mineral acid, or with acetic acid if alkaline, treated with 10 c.c. of glacial acetic acid and 2 c.c. of 30 per cent. ammonium molybdate solution, and titrated with standard potassium ferrocyanide solution until a persistent yellowish-pink coloration forms.

In the case of a zinc ore, 1·5 grms. of the finely powdered material are treated in a roomy dish with 20 c.c. of *aqua regia*, evaporated to dryness, and the dry residue mixed with 15 c.c. of concentrated hydrochloric acid and evaporated to dryness to render the silica insoluble. The residue is boiled with 5 c.c. of hydrochloric acid and 5 c.c. of water, and the liquid then diluted with 200 c.c. of water, treated with hydrogen sulphide to precipitate the heavy metals, and filtered into a large beaker. The excess of hydrogen sulphide is expelled by boiling, the solution boiled with a few drops of nitric acid and then treated with ammonia, ammonium carbonate and a small quantity of hydrogen peroxide. The precipitate is filtered off, washed into a 500 c.c. flask and redissolved in boiling dilute hydrochloric acid, the solution then evaporated to expel most of the acid, and the residue, dissolved in water, and the solution treated, drop by drop, with ammonia until precipitation is complete. Both liquid and precipitate are introduced into the 500 c.c. flask containing the previous solution, and the whole cooled and made up to the mark. After filtration 100 c.c. are evaporated to about 25 c.c., and the procedure described above then followed. T. H. P.

Electrometric Titration of Chromic Acid. H. T. S. Britton. (*J. Chem. Soc.*, 1924, **125**, 1572–1582.)—It has been found possible to titrate chromic acid electrometrically provided that certain precautions are taken. The electrode must be of platinum foil, not wire, should contain lead and be coated with platinum black, and should be immersed completely in the perfectly still solution. It is, however, not easy to obtain concordant results, so that an oxygen electrode is preferable, this being standardised against the hydrogen one. The course of the neutralisation curve shows that there are two stages—the formation of sodium hydrogen chromate, then the extremely weakly dissociated anion $HCrO'_4$, which is neutralised to form sodium chromate. The dissociation constant of $HCrO_4$ is $4·4 \times 10^{-7}$ at 18° C., whereas H_2CrO_4 is almost completely dissociated. For the titration with an oxygen electrode the chromic acid solution are connected through a bridge with saturated potassium chloride and a normal calomel cell, and the P.D. between it and the electrode is measured potentiometrically in the usual way. The E.M.F. obtained is more steady when oxygen is passed through at a fairly uniform rate of about 5 bubbles per second. H. E. C.

Colour Reaction for the Detection of Zirconium and Fluorine. J. H. de Boer. (*Chem. Weekblad*, 1924, **21**, 404–405.)—Many metals, under given conditions, yield colour reactions with alizarine sulphonic acid, but it was found that, in the presence of a very large excess of concentrated hydrochloric acid only the yellow colour due to zirconium persisted. It was possible by means of this reaction to detect 0·02 mgrm. of zirconium per c.c. The change to the yellow colour is effected particularly rapidly in the presence of small quantities of fluorine ions. By mixing 2 c.c. of a zirconium solution containing 0·01 grm. zirconium per c.c. with 5 c.c. of an alizarine sulphonic acid containing 0·003 grm. per c.c., and adding 60 c.c. of concentrated hydrochloric acid, a reagent is obtained capable of detecting 0·001 mgrm. of fluorine in 1 c.c. water, whilst, if double the amount of concentrated hydrochloric acid is used, the sensitiveness of the reaction is increased ten-fold. W. S. S.

Microchemical Estimation of Perchlorate. A. Pamfilov and W. Jofinow. (*Chem. Zeit.*, 1924, **94**, 541.)—The solution under examination is treated with solid sodium bicarbonate till just alkaline; 3 to 4 drops are placed on a slide, treated with 10 to 15 small crystals of rubidium chloride and dried carefully but completely over a flame. The crystalline mass is almost dissolved in 1 or 2 drops of weak potassium permanganate solution and the liquid evaporated till crystals begin to form on the circumference. The subsequent crystallisation is observed under the microscope. The development of isomorphous rose-coloured rhombic crystals denotes the presence of perchlorate. These are easily distinguished from the small deep coloured crystals of permanganate, which, as a rule, are absent in the presence of perchlorate if the permanganate solution has been weak enough. This method makes possible the detection and, with practice, an approximate estimation of perchlorate in solutions of 0·1 per cent. strength. It is more delicate than the Klobbie-Visser method, because of the greater solubility of the potassium perchlorate crystals. It can be used successfully in the presence of considerable proportions of chloride and chlorate. R. F. I.

Physical Methods, Apparatus, etc.

A simple Non-splash Ring for use with Scheibler's Desiccator. S. C. Bradford. (*J. Chem. Soc.*, 1924, **125**, 1546.)—This consists of a bell-shaped ring to rest, narrow end downwards, on the constriction of the acid reservoir. D. G. H.

Reviews.

THE SIMPLE CARBOHYDRATES AND THE GLUCOSIDES. By E. FRANKLAND ARMSTRONG, D.Sc., Ph.D., F.R.S., F.I.C. (Monographs on Biochemistry, edited by Dr. R. H. A. Plimmer and Dr. F. Gowland Hopkins.) Fourth Edition. Pp. xii.+293. London: Longmans, Green & Co. 1924. Price 16s. net.

The present edition of this work, which is larger than its predecessor (ANALYST, 1919, **44**, 427) by over fifty pages, bears abundant evidence of the rapid progress in our knowledge of the carbohydrates and their derivatives during the past five years. The enlargement of the book is almost entirely due to the amplifications and additions rendered necessary by recent research.

Dr. Armstrong devotes a considerable amount of attention to the active forms of the hexoses now frequently referred to as the γ-forms, which appear to be of the utmost importance from the biochemical point of view. The constitution of these isomerides is still in question. Thus γ-glucose, until recently considered to contain a 1:2 ethylene oxide ring, is now thought to contain a propylene oxide ring. It is much more reactive than the well-known butylene oxide (α- and β-) forms of glucose, and it has been suggested that the occurrence of an excess of sugar in the blood in cases of diabetes is due to failure to convert the comparatively inert butylene oxide form into the active γ-form. The active or γ-form of fructose is provisionally regarded as containing an amylene oxide ring; this form of fructose is perhaps present in sucrose.

A comparison of the chapter on the disaccharides (Chap. V.) with the corresponding chapter in the preceding edition illustrates recent progress in the chemistry of the sugars in a very striking manner. The work of Irvine and his collaborators on the methyl derivatives of the sugars has continued to bear remarkable fruit. Thus, in the cases of maltose, cellose and gentiobiose and the trisaccharide, raffinose, the nature of the union of the hexose residues has been elucidated.

The still more formidable problems involved in the investigation of the constitution of the polysaccharides are dealt with in a chapter which is a new feature of this edition. Here, again, the record is one of rapid advance, an advance which is largely owing to the work of the Purdie-Irvine school. It is interesting to note that inulin, which yields fructose only on hydrolysis, is derived from the γ-form of this sugar.

The present edition maintains the very high standard of the earlier ones, and Dr. Armstrong is to be congratulated on the discrimination shown in his selection of the new material now incorporated in the book. LEWIS EYNON.

INKS: THEIR COMPOSITION AND MANUFACTURE. 3rd Edition. By C. AINSWORTH MITCHELL, M.A., F.I.C., and T. C. HEPWORTH. Pp. xvi.+336. London: Charles Griffin & Co., Ltd. Price 12s. 6d. net.

Although the use of ink is almost universal and the amount consumed considerable, yet any intimate knowledge of it is very uncommon and, from the manner in which it is generally referred to, it might be thought that ink is always a fluid which is used only for writing, usually of a black colour, though occasionally red, and always of approximately the same composition. A reference to the table of contents of the book under review is sufficient to correct any such idea, and it is seen that ink may be in a state of powder, paste or liquid, and of almost any colour and composition, and that it is employed not only for writing, but also for printing and marking. An account therefore of the history, uses, modes of manufacture, properties, and methods of testing of the various kinds of ink, written by a specialist, must needs be both interesting and valuable. That this is so is proved by the fact that the present is the third edition, the first having been published in 1904. The present edition, which embodies all the latest knowledge on the subject of inks, is an enlargement and revision of the earlier work, and every section has been brought thoroughly up to date.

The book, which opens with a short historical introduction, is divided into three sections, namely, Writing Inks, Printing Inks and Inks for Miscellaneous Purposes respectively, the latter including Copying Inks, Marking Inks, Safety Inks, Sympathetic Inks, Ink Powders and Tablets, Stencil Inks, Machine-ruling Inks, Inks for Rubber Stamps, Inks for Typewriters, Waterproof and Fireproof Inks, and Inks for Writing on Glass, Metal, Wood, Leather, and Ivory. The book concludes with a Bibliography, a List of English Patents for Inks, extending from 1688 to 1923, and an excellent Index.

The book will prove invaluable to the manufacturer, who desires for his product satisfactory quality and uniformity of composition; to the analyst who may be called upon to test supplies of the raw materials or of finished inks in order to ascertain whether they conform to specification requirements, whether they are worth the price asked and whether they are suitable for the purpose required; to the specialist who wishes to prove the kind or date of an ink on a document suspected to be forged and to the users of the many different kinds of ink enumerated.

While all sections of the book should appeal to chemists, those that will prove most useful are the ones dealing with the chemical composition and methods of examination of the raw materials (including a very complete account of the different tannins, their nature, properties and methods of estimation), the nature of the compounds formed during the manufacture of iron-gall ink and the changes which occur on keeping, the composition and tests for the various colouring matters, particularly the organic ones, employed for making coloured inks, and the chemical examination of ordinary writing ink, both in bulk and also in the form of writing on a document.

Throughout the book there will be found evidence that it is no mere compilation of the work of others, but that it incorporates the results of much practical knowledge and original research on the part of the senior author, who is responsible for the chemistry. The reviewer from personal experience can bear testimony to the value of the book.

One is accustomed to find the books produced by this firm of publishers very well printed on good paper and with excellent reproductions of the illustrations, and the present volume forms no exception to the rule. A. LUCAS.

OLII E GRASSI. VEGETALI, ANIMALI E MINERALI. 2nd Edition. By G. FABRIS. Pp. 671. Milan: U. Hoepli. 1923. Price 24 lire.

This is one of the useful little volumes in the series of industrial manuals which this leading firm of Italian publishers has been producing for some years, and the name of its author is well known as that of an authority on the chemistry of oils and fats.

The arrangement of the book is similar to that of the last edition, the first section dealing with vegetable oils, the second with animal oils, and the third with mineral oils. In each case the methods of extraction and purification of the respective oils and their composition and examination are discussed, and tables of the principal analytical values of 470 different fatty oils and fats, occupying 55 pages of the book, are included.

A good but brief outline of the methods of fat analysis is given, although this needs supplementing in some places. It is interesting to note that, according to the author (p. 182), Hübl's method of estimating the iodine value is still the one most generally used in Italy.

On p. 187 the measurement of heat of bromination as a rapid analytical test is attributed to Marden (1916). The reviewer has for years been under the impression that the test was devised by Hehner and Mitchell, and that it was first published in THE ANALYST.

For English chemists the most valuable portions of the work are the descriptions of the methods and plant now used in Italy for the production of olive oil, and the statistics of the Italian oil industry. The book concludes with a fairly full bibliography, but lacks an index. The classified table of contents can hardly be regarded as an efficient substitute for one. EDITOR.

PERFUMES AND SYNTHETICS WITH ESPECIAL REFERENCE TO SYNTHETICS. By W. A. POUCHER. Pp. xi.+462. London: Chapman & Hall, Ltd. 1923. Price 21s.

Of the more recent books on perfumery and allied subjects this is certainly one of the best; indeed, a perusal of the preface renders criticism difficult. The division of the subject matter into parts has strengthened the book in many ways. The first part is somewhat scrappy, but fairly comprehensive, though the absence of chemical formulæ indicative of the constitution of the various synthetic odorous substances is a matter for regret. The synthetic esters would have been better dealt with had the physical and chemical data been included in the descriptions of their properties; after all, it *is* chemistry that is concerned with their manufacture, and, alas, their adulteration, and every user of them, sooner or later, needs exact chemical knowledge of their composition.

An admirable feature of the book is the photographic illustration of processes and plant, though here, again, details of stills and the like might have been given in the form of a working drawing, rather than merely as a picture. A few of the pictures seem familiar.

The chapter on "Fixation" may be singled out as one of the best in the book, and the author is to be congratulated on this lucid and practical account of a very important branch of the perfumer's art. The monographs on floral perfumes are altogether too brief and should, in the writer's opinion, have been enlarged so as to aid the chemist who may have to examine such costly substances; really, though it may appear ungracious to say so, legend might have given place to practical details.

The pages relative to compounding preparations are full of good things, and the information is on the whole reliable and, in the main, modern. The style of the volume is excellent, and publisher and author alike must be congratulated on the production of a book which must be in the library of everyone interested in this subject. L. G. RADCLIFFE.

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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

The Hon. Secretary wishes to direct special attention to the notice on page 556.

Obituary.

OTTO HEHNER.

It is difficult to realise that Hehner is gone from us. He was so long and so intimately connected with our Society that it is only gradually we shall come to know the full extent of our loss. The blow is all the more severe because so unexpected. Only a few days before his death he had written to say that the end of his work in South Africa was in sight, and that he was intending shortly to return to London. It was not to be, for he was attacked by malaria and died on September 9th.

Otto Hehner was born on November 25th, 1853, at Marienberg, in Nassau. His father was a judge of the High Court of Appeal in Wiesbaden, and it was doubtless this legal strain in his blood which gave him that intuitive grasp of the legal aspects of a case, which was freely acknowledged by the leaders of the English Bar, who so often met him in consultation. He was educated at Wiesbaden and studied chemistry under Fresenius, whose laboratory was already recognised as the leading training ground in Germany for the profession of analytical chemistry. He was for a time assistant to Fresenius, until, in 1873, he came to this country to become assistant to Professor Bischof at the Andersonian College in Glasgow. At about the same time Ramsay was also starting his professional career as assistant to Bischof, and the association between the two young men ripened into a close friendship which only closed with Ramsay's life.

Only a few months ago Hehner gave the writer a humorous description of his early experiences in Glasgow and of his ineffectual attempts to adapt himself to the requirements of a Scottish Sabbath, more stringent then than now.

From the very outset he had been attracted to the chemistry of food and the problems of preventing its adulteration. The Act of 1872, which led to the appointment of Public Analysts, had only recently come into force, and Hassall, to whose labours this Act was largely due, was accepted as the principal authority on the subject. He invited Hehner to join him as one of his chemical assistants, and early in 1874 Hehner left Glasgow for Ventnor, where Hassall had established his laboratory. Here he had as fellow assistant Mr. Arthur Angell, who is still with us. In addition to carrying out the chemical routine work of the laboratory, the two young assistants engaged in research work, and it was then that Hehner discovered the essential difference between butter fat and the animal body fats, and devised that process which will always be associated with his name—the determination of the *Hehner value.*

The same year Dr. Hassall, giving evidence before a Parliamentary Commission to enquire into the working of the Act of 1872, admitted that he did not know of any method of detecting foreign fat in butter, but stated that his assistant, Mr. Otto Hehner, claimed to have discovered a method.

The findings of this Commission led to the passing of the Sale of Food and Drugs Act of 1875, which made the appointment of Public Analysts no longer permissive, as in the 1872 Act, but compulsory. The various counties and boroughs then had to discover suitable men for the purpose, and many solved the difficulty by appointing their medical officers, most of whom knew but little chemistry.

In 1877 Hehner came to London, and shortly after started in practice as an analytical and consulting chemist, and the reputation which he had already gained as a scientific authority on butter helped him in establishing this.

In 1878 he was appointed Public Analyst for the Isle of Wight, an appointment which he retained until 1909. The same year he received the appointment for Ryde, which he only resigned three years ago.

A few years later other public appointments followed:—Derbyshire (1881–1893); Derby Borough (1881–1916); Nottinghamshire (1885–1921); and West Sussex (1890–1921); and, after the passing of the Fertilisers and Feeding Stuffs Act, he became Agricultural Analyst for the Isle of Wight (1893–1909) and for West Sussex (1893–1921).

The Society of Public Analysts was founded in 1874, and Hehner joined it in 1876 while still at Ventnor. After coming to London, he took an active part in the work of our Society. He was first elected a member of the Council in 1880, and two years later became one of the Secretaries, which office he resigned on his election as President in 1891. Since then he was continuously on the Council as Past President, and, until the war, seldom missed a meeting.

He was an abstractor of THE ANALYST for some years, and edited the journal for a month after the sudden death of Dr. Sykes, in 1907, and he continued to be a member of the Publication Committee until the present year.

Most of his original work was concerned with the composition and analysis of food and drugs, and was mainly read before this Society and published in our journal. A reference to the decennial indexes will show the wide scope of this work, which included researches into the chemistry of fats, methods for the examination of meat extracts, the analysis of honey, experiments on the action of preservatives, the adulteration of beer, the analysis of vinegar, various contributions to the analysis of milk and butter, water analysis, etc., etc. His first paper published in THE ANALYST was, appropriately, one on butter analysis in collaboration with Angell (ANALYST, 1877, 1; 147); his last paper was one on the estimation of pentosans in collaboration with W. P. Skertchly (ANALYST, 1899, 24, 178), but he continued to take an active part in the discussions at our meetings until 1914. His alcohol tables, first published in the fifth volume of THE ANALYST, and afterwards produced in book form, supplied a great want and are still used. He was also responsible for the article on "Adulteration" in the ninth edition of the *Encyclopædia Britannica*—an admirable digest of the subject, in which, again, his legal acumen is clearly shown.

Among the outstanding investigations associated with his name it may be recalled that the method of analysing beeswax, based upon the constant ratio of free cerotic acid to myricin, was first devised by Hehner in 1881 (ANALYST, 1881, 8, 16), and that Hübl, whose name is associated with the method, did not publish the same discovery until a few months later. The dichromate method of estimating glycerol in fats was another contribution to analytical chemistry and has long been accepted as a standard process (ANALYST, 1888, 12, 44), and the Hehner method of estimating the hardness in water without the use of soap solution is almost as widely known (ANALYST, 1884, 8, 77).

Hehner was an original member of the Society of Chemical Industry, and subsequently became Chairman of the London Section. In 1878 he was elected a Fellow of the Institute of Chemistry, and he served as Vice-President on three occasions, the last being 1915–1918; he was an examiner from 1895 to 1899, and a Censor from 1901 to 1903.

Within a few years he had come to be generally accepted as one of the leading authorities on all chemical questions affecting the public health, and his help was sought by many public authorities in getting their water bills passed through Parliament. In 1901 he acted as Chairman of the Joint Committee of our Society and the Society of Chemical Industry on the methods of estimating arsenic, and he was also Chairman of the Expert Committee of 1909 to 1911 and that of 1919 to 1921, called together to standardise the methods of glycerin analysis.

In 1901 a Parliamentary Committee was set up to inquire into the use of preservatives and colouring matters in foods, and he was one of the most important witnesses at that abortive inquiry.

The busy years swept on, their days filled with a never-ending round of consultations, legal cases, committee meetings and meetings of scientific societies, not to mention the constant supervision of his extensive routine laboratory practice and the investigation of chemical problems requiring experimental solution, until, in 1913, he was at the summit of a successful professional career. In November of that year he invited his past and present assistants to a dinner to celebrate his sixtieth birthday. He was in one of his gayest and happiest moods, full of plans for the future, and the prospect for his remaining years seemed unclouded. And then, within a year, came the war.

His was one of the great personal tragedies of that catastrophe. Over and over again he had impressed upon his friends in Germany that the spirit of domination which had developed there since his boyhood was essentially parochialism, and was leading that country straight to disaster. When the inevitable crash came he at once ranged himself actively on the side of his adopted country, where he had long been naturalised, and he could not understand why the average Englishmen should have hesitated to accept him as one of themselves.

Those who knew him never wavered in their attitude of friendship and trust towards him throughout that trying time, and it is significant of the esteem in which he was held throughout the country that he was unanimously chosen as the official referee for glycerin contracts between the War Office and the soap manufacturers. He also worked continually in connection with the control of foods, serving as the Chairman of two Committees on the standardisation of the methods of analysing oils and fats, and he was one of the first to make experiments to discover the best type of mask to afford protection against poison gas.

He never spared himself in his efforts to help this country, and for all this work he refused to accept any remuneration whatsoever. The special letter of thanks for his services during the war, sent to him by Lord Moulton, brought some comfort to him, but he was deeply hurt by the attitude taken up by certain sections of the press and some public authorities. He failed to make allowance for the loss of a sense of proportion in those who, egged on by the sensation-sowing papers and eager to express their active detestation of the methods of atrocity introduced into the war, often became unjust to the individual of German antecedents.

And thus it was that after the war, while he was even more busy than before, he had lost some of that joyous zest in his work which was so characteristic of the man. He could not altogether shake off the memories of those four years, and he, therefore, in 1921, welcomed the chance of visiting South Africa. When he returned last year he actively resumed his professional work, and later in the year was chosen as the representative of our Society on the new Parliamentary Committee on Preservatives. Being obliged to return to South Africa for a time, he resigned from this Committee, but his public work continued, for his help had been welcomed by the South African Government, and he had been chosen a member of a Committee appointed to consider the question of power alcohol (1922–1924).

The characteristics which must have struck everyone first in dealing with Hehner were his straightforwardness, his love of justice, and the simplicity of his nature. To those who did not know him well his manner might seem brusque at times, and he was inclined to be impatient with those whose brains moved more slowly than his. Yet these were only surface faults, and beneath was a deep vein of genuine sympathy. None of his friends ever turned to him in vain for counsel or help, and only those who were closely associated with him know of his many acts of kindness and generosity even to complete strangers. His outlook on life was shrewd, but never cynical, and was always instinct with a broad humanity. When the writer first knew him he was inclined to be dogmatic in the expression of his views, but time softened this, and in his later years he showed a kindly tolerance of the opinions of those from whom he differed.

In his chemical work he showed that gift which is akin to genius, of seeing instinctively the crux of a problem, and he would eagerly devise ingenious methods of testing the truth of his hypothesis. At the same time he had the drawback, which is frequently part of such a temperament, of being too readily discouraged if his first efforts were unsuccessful.

Much of his time was spent in the Law Courts, and he was never happier than when he was working up the chemical side of a case and devising experiments which could be shown in Court to convince judge and jury of the truth of his contention. He was an excellent witness, clear and concise in his evidence, and always ready with a humorous answer for a cross-examining counsel.

A keen lover of nature, Hehner delighted in long country tramps with one or more intimate friends, and in the interludes of conversation, which would range over an infinity of topics from the trivial to the profound, he would be on the alert for familiar plants, and would note the habits of each wild animal or bird met with on the way. On one of his last walks with the writer he raised the question of how long a soaring lark could remain in the air, and with characteristic eagerness at once put the matter to the test.

Although in the later years of his life he was denied the happiness of the home life for which he was so well fitted, he remained unsoured, and was always ready to join in any piece of boyish fun on hand. In one of his letters to the writer he mentioned how happy he had been in his friendships throughout life. He was a lovable man, and his death makes a great and lasting gap in the lives of all of us who loved him.

C. A. MITCHELL.

The Quantitative Estimation of the Degree of Hydrolysis of Gallotannin by Tannase.

BY WINIFRED NELLIE NICHOLSON, B.Sc., AND DONALD RHIND, B.Sc.

(*Read at the Meeting, October* 1, 1924.)

THE main function of tannase is the disintegration of the gallotannin molecule, gallic acid being produced. With the view of measuring the activity of tannase a quantitative method was elaborated in this laboratory by Rhind and Smith (*Biochem. J.*, 1922, **16**, 1), which gives the amount of unchanged gallotannin left after hydrolysis by tannase. The gallotannin is estimated by the caseinogen absorption method (Nierenstein, *Chem. Zeit.*, 1911, **36**, 31), as modified by Spiers (*J. Agric. Sci.*, 1914, **6**, 77) in this laboratory for the estimation of small quantities of gallotannin.

During the course of our work on tannase, however, it became necessary also to estimate the amount of gallic acid produced by the tannase. Such a method has been described by Freudenberg and Vollbrecht (*Zeitsch. physiol. Chem.*, 1921, **116**, 277). In this method the gallic acid formed by the action of tannase on gallotannin or methyl gallate is estimated by titration with sodium hydroxide, litmus paper being used as an indicator. It is assumed by Freudenberg and Vollbrecht that, under the conditions chosen by them, only the carboxyl group in gallic acid reacts with sodium hydroxide. This method has been investigated by one of us (D.R.), but has been found unreliable.

On the other hand, concordant results have been obtained by one of us (W.N.N.) when using Mitchell's colorimetric method for the estimation of small quantities of gallic acid. (ANALYST, 1923, **48**, 2.) This method, slightly modified so as to adjust it to our requirements, has been in constant use in this laboratory for the last eighteen months, and has been found to be trustworthy.

In the present communication we give our results with (1) the method of Freudenberg and Vollbrecht, and (2) the modification of Mitchell's method, as applied to the hydrolysis of gallotannin by tannase.

I. THE METHOD OF FREUDENBERG AND VOLLBRECHT.—The following is a brief summary of the results obtained:—(1) Under the exact conditions laid down by Freudenberg and Vollbrecht, namely: (*a*) solutions of the same strengths as theirs, *i.e.* $N/40$ sodium hydroxide solution and 0·333 per cent. gallic acid, (*b*) Freudenberg and Vollbrecht's correction for gallic acid, and (*c*) their correction for distilled water, there were errors varying from +2·8 to −3·8 per cent. In view of the fact that always the same amount of gallic acid was used for these experiments, this error, which varied in both directions, is obviously too great. (2) The use of litmus paper as an indicator, as recommended by Freudenberg and Vollbrecht, was found to have the disadvantages that the end-point is difficult to see, and that its appearance depends upon the size of the drops of solution placed upon the paper, the rate of absorption of the solution, the light, and the depth of colour of the paper used. (3) Experiments using bromthymol blue and phenol red as internal indicators gave the following results:—(i) *Bromthymol Blue.*—The bluish-green colour was taken as the end-point, although it gave uniformly high results. The correction required is 0·45 c.c. of N/40 sodium hydroxide solution for 20 c.c. of a 0·333 per cent. solution of gallic acid. By using three drops of the indicator and titrating in good daylight (electric light was found to be unsuitable) the errors lay between +0·4 and −1·7 per cent. Although bromthymol blue is an improvement on litmus paper, when titrating gallic acid by itself, the method fails in the presence of tannase, as errors of +3·8 to −1·4 per cent. were obtained. "Killing" the tannase by boiling the solution does not reduce the error. (ii) *Phenol Red.*—A correction of 0·92 c.c. of $N/40$ NaOH was required. The errors varied between +0·3 and −0·4 per cent., and this error remained the same in the presence of tannase. Diastase* and emulsin increase the error to −2·6 per cent. Phenol red fails, however, entirely when gallotannin is replaced by methyl gallate and ethyl gallate, as used by Freudenberg and Vollbrecht, the average error then rising to +50 per cent.

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* From an investigation carried out by Mr. C. J. Pool in this laboratory it appears that diastase and invertase, in addition to other enzymes, are present in the mycelium of *Aspergillus niger*, which was the source of the tannase used in our work.

We therefore discarded the Freudenberg and Vollbrecht method and the attempted modifications of it.

II. Mitchell's Method.—The method described by Mitchell for the estimation of gallic acid, is a colorimetric one, based on the fact that the acid gives a bluish violet-colour with iron salts. Mitchell found that the most stable colour is produced if a mixture of ferrous sulphate (0·1 per cent.) and Rochelle salt (0·5 per cent.) is used as the reagent. The estimation is carried out by means of a colour comparison in Nessler tubes in the usual way.

In using Mitchell's method in this work, slight modifications were introduced. In preliminary experiments it was found that the error, when comparisons were made in daylight, lay between −2·59 per cent. and +4·85 per cent. and other estimations were therefore carried out by artificial light. When ordinary electric light, shaded by white paper, was used, the errors were between −1·81 and +4·06 per cent.; electric light shaded by light blue paper gave errors of −4·31 to +7·97 per cent.; when the light was shaded by dark blue crinkled paper, the errors were reduced to between −1·33 and +2·90 per cent., the average of seven readings being +0·83 per cent. The last modification was therefore adopted.

In order to facilitate the making of colour comparisons by artificial light during the day, the apparatus shown in the diagram, was designed:

An oblong wooden box (18 in. by 7 in. by 7 in.), painted black inside, and having a plate-glass lid, was fitted with an electric bulb as near the centre as possible. On this, in the centre, was placed a second box (5 in. by 7 in. by 10 in.), into which were fitted two Nessler tubes, their taps projecting through a slit in each side, and the tubes themselves extending about 1 in. above the lid through two holes in the top. This box had no base, so that, when in position, the tubes stood on the plate glass, immediately above the light. The remainder of the plate glass was covered with two movable pieces of wood, whilst the part in the centre, beneath the tubes, was covered with dark blue crinkled paper. The whole of the inner surface of both boxes was blackened, so as to prevent reflection as far as possible. The taps were connected by rubber tubing with a sink, so that it was impossible to tell how much solution was being run out. The use of this apparatus thus tends to eliminate personal error.

a Blue. *b* Plate glass. *c* Electric bulb.

The unchanged gallotannin was precipitated with quinine hydrochloride, as recommended by Mitchell. The small quantities of quinine tannate thus obtained were colloidal and difficult to filter. Sodium chloride was therefore added, and the solution then yielded a clear filtrate on shaking or centrifuging. It was, therefore, essential to find how far the presence of an excess of quinine hydrochloride or of sodium chloride affected the colour comparisons. It was found that, whereas quinine hydrochloride has no effect, the presence of salt introduces a constant plus error, and in order to eliminate this error, the same amount of salt was added to the standard tube in each estimation.

Finally the following technique was adopted:—The hydrolyses were carried out in a series of test tubes, into each of which were put 10 c.c. of a gallotannin solution (containing about 3 grms. per litre). To this was added 1 c.c. of mycelium extract (prepared by shaking 1 grm. of mycelium powder with 50 c.c. of distilled water for 3 hours, allowing this to stand for 21 hours, and filtering). A layer of benzene was poured on the surface to prevent the growth of fungi. The test tubes were corked tightly and placed in an incubator at 25° C., one being used for a gallic acid estimation every twenty-four hours. To precipitate the unchanged gallotannin, 4·5 c.c. of a 1 per cent. solution of quinine hydrochloride were added (this being an excess), and 0·5 c.c. of a 16 per cent. solution of sodium chloride, to coagulate the precipitate. One c.c. of this mixture was added to one of the Nessler tubes, and 1 c.c. of standard gallic acid, and 1 c.c. of a 0·5 per cent. solution of sodium chloride to the other. To each tube 2 c.c. of Mitchell's reagent were added, the volume in each case made up to 100 c.c. and the liquid stirred. (It was found convenient to use a glass rod with a flattened knob at the base for stirring.) The darker liquid was then run out until the colours approximately matched, and a sufficient quantity of Mitchell's reagent was added to make up for the amount run out, as unless the concentration of the reagent was the same in both tubes, different colours were produced. The volume was again made up to 100 c.c., the liquid stirred, and the colours now matched as accurately as possible.

We wish to thank the Department of Scientific and Industrial Research for grants which have enabled us to carry out this investigation. Our thanks are also due to Dr. Nierenstein for the interest he has shown in the work and the advice he has given.

BIOCHEMICAL LABORATORY,
UNIVERSITY OF BRISTOL.

DISCUSSION.

Dr. M. NIERENSTEIN, who communicated the paper in the absence of the authors, said that Mitchell's method had not only proved of help in connection with the work on tannase, but had also been of the greatest value in other work on the tannins which was in progress in his laboratory. Thus it seemed to offer a means to the solution of the vexed question whether glucose was an essential part of the gallotannin molecule. From a series of experiments which were in progress in his (Dr. Nierenstein's) laboratory it was evident that yeast decreased the glucose content of gallotannin which had been purified by Fischer's method. This decrease in glucose, however, was *not* accompanied by an increase of free gallic acid, as was to be expected from the Fischer formula of gallotannin. This type of work could only be carried out with the aid of Mitchell's method. It must be emphasised that these observations confirmed the views held by him during the last 20 years, namely, that glucose was not an essential part of gallotannin. In this connection it must be remembered that Mitchell had described a gallotannin which was practically free from glucose, and that this had also been confirmed by him (the speaker).

Mr. C. A. MITCHELL said that he was pleased to have the value of his method confirmed by independent workers. He had sent to Bristol a specimen of that remarkable glucose-free tannin in which he had found 10·5 per cent. of gallic acid (ANALYST, 1923, **48**, 9), and Miss Nicholson, working under her own conditions, had found it to contain 10·7 per cent. An objection urged against the method was that the comparison ought to be made under definite hydrogen ion concentrations. So far as his experience went a good deal of latitude was permissible in this respect; all that was necessary was to neutralise the acidity of the ferrous sulphate. The point was now under investigation at University College, Exeter, and he hoped that, before long, standard conditions for the test might be established. He, too, had experienced the difficulty of filtering the colloidal solutions, but had overcome it by adding a little Spanish clay to the liquid. This avoided the introduction of a soluble salt which had some effect upon the reaction.

Mr. A. CHASTON CHAPMAN remarked that the subject of the constitution of gallotannic acid was in the nature of a hardy perennial, and stated that although gallotannic acid might possibly—as was sometimes asserted—be an anhydride of di-gallic acid, yet he thought that the authors' results need not necessarily be regarded as entirely disproving its glucosidal character. The action of tannase was apparently different in some respects from that of dilute acids, and in the attack by yeast it was quite possible, having regard to the large number of enzymes secreted by that organism, that the gallic acid might itself be decomposed. It might very well be that the true gallotannin was, in fact, a glucoside, and that the product which did not yield glucose was a simpler derivative of it. It might, in other words, perhaps be, after all, a question of definition. They were very greatly indebted to the authors and to Dr. Nierenstein, under whose direction the work had been carried out, but he thought that they would agree with him that the last word had not yet been said on this subject.

Preliminary Notes on the Composition of the Fat of Goats' Butter.

BY FRANK KNOWLES AND JOHN C. URQUHART.

(*Read at the Meeting, October* 1, 1924.)

AT the present time goat-keeping in this country appears to be increasing and, with a vigorous movement being carried on to popularise it, may become an industry of some importance. As is well known, this "poor man's cow" is a very hardy animal, and under reasonable conditions will produce milk as economically as the average cow; and, generally speaking, the milk is of richer quality both in fat and solids-not-fat. There are to-day in Essex herds of goats which yield, on the average, approximately a gallon of milk per day per head, and this figure is often exceeded. A considerable amount of this milk is churned into butter which is sold as "butter"; the owner of quite a small herd of goats recently informed us that he was selling a surplus over his own requirements of 10 lbs. of "butter" per week.

The prejudice against goats' milk or butter on account of alleged objectionable taste is rapidly disappearing, there being no real grounds for this prejudice if the goats are kept under proper conditions. Certainly, those goat-keepers making butter have no difficulty in disposing of their product.

The important question may be asked: Is it legal to sell this product as butter? If answered in the affirmative the point could be raised: Do the analytical standards of purity which are in use for butter apply? If a negative reply be given, what standards should be fixed for goats' butter?

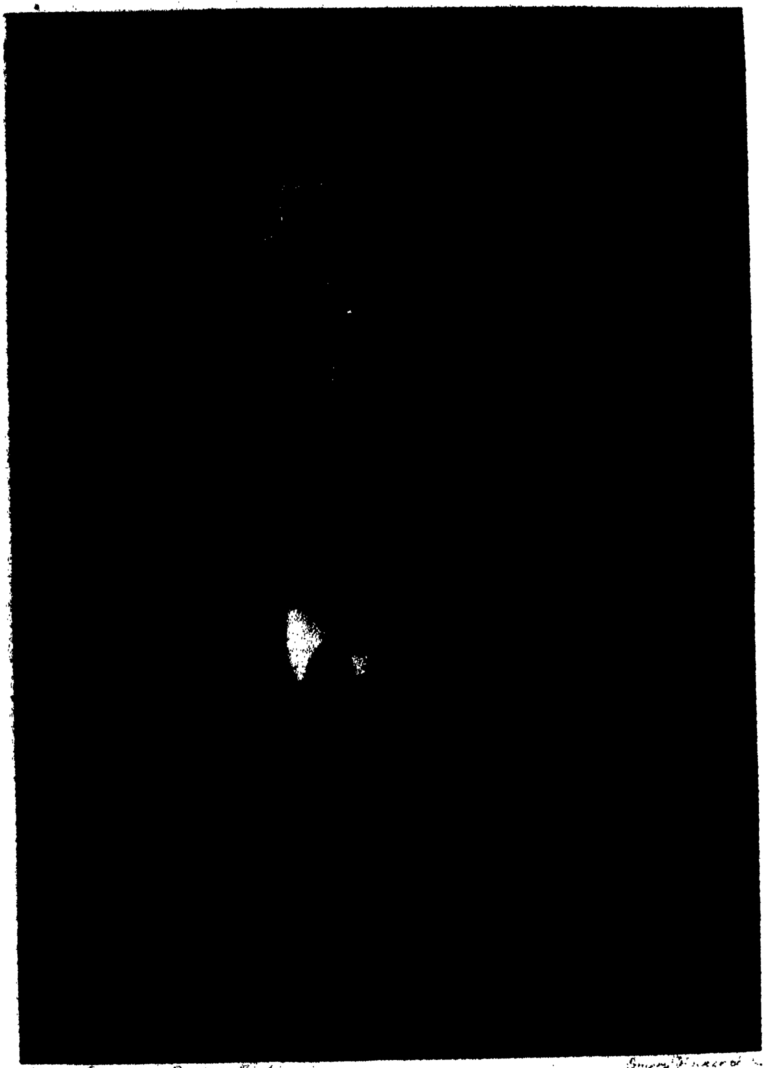
The definition of "butter" in the Margarine Act 1887 (50 & 51 c. 29) is as follows:—"The word 'butter' shall mean the substance usually known as butter, made exlusively from milk or cream, or both, with or without salt or other preservative, and with or without the addition of colouring matter."

The Butter and Margarine Act, 1907 (7 Edw. 7, c. 2), describes milk-blended butter as "any mixture produced by mixing or blending butter with milk or cream (other than condensed milk or cream)." Obviously this definition of butter and also, presumably, of cream depends upon the interpretation which is put upon the word milk.

In this connection an extract may be quoted from *The Law Relating to the Sale of Milk*, published by Messrs. Bibby & Sons:—"So far as we know, there is no exact definition of milk on our statute book, but the Congress of Geneva framed the following: 'Milk is the integral product of the entire and uninterrupted milking of a female cow in good health, and well nourished and not overworked. It ought to be collected in a proper manner and contain no colostrum.' "

That this is the generally accepted definition of milk is supported by the Milk and Dairies Act of 1915 and that of 1922, which will come into operation in September, 1925, which ignores the existence of any milk other than that of the cow. Paragraph 5 of the Act of 1922 prohibits the sale of milk of a cow suffering from tuberculosis of the udder, but it will presumably be quite lawful to sell the milk of goats where such animals are similarly infected.

If this definition be accepted, it is clear that the milk or butter obtained from the goat is not correctly named if sold, respectively, as "milk" or "butter." As, however, goats' milk and cows' milk are not easily distinguishable, this misnaming may occur, either through ignorance of the law, or for the purpose of effecting a sale of goats' milk to persons who might, conceivably, be chary of purchasing it if it were so named. The possibility of cows' milk and goats' milk being mixed and sold to dairies for butter and cheese-making must also not be overlooked. It is, therefore, very desirable to ascertain if differences exist between the customary analytical standards for cows' butter and the values obtained by using the same methods of analysis on a large number of samples of butter which are known to have been made from goats' milk only. Further, if differences are found consideration could be given to the desirability of setting up similar standards



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Otto Hehner

for goats' butter to which such butter should comply if adulteration is not to be considered established or to be presumed.

The mean analytical values from closely agreeing duplicates obtained by us when working with the fat of genuine goats' butter obtained direct from owners of herds are given below. The methods of analysis used are the usual ones; care being taken to follow closely the details in the case of such conventional methods as the Reichert-Wollny and Polenske values, etc.

ANALYSES OF FAT OF GOATS' BUTTERS.

Sample Ref.	Reichert-Wollny value.	Polenske value.	Kirchner value.	Refractive index.	Iodine value.	Sp. Gr. at 100° F./100° F.
1	27·66	8·65	—	1·4554	24·73	0·9326
2	25·08	7·50	—	1·4554	28·92	0·9209
3	27·77	7·85	16·82	1·4554	33·56	—
4	27·22	7·00	18·56	1·4547	27·92	—
5	25·96	8·70	17·20	1·4547	34·84	0·9346
6	24·47	5·30	17·12	1·4554	34·03	0·9218
7	26·07	7·6	18·04	1·4552	30·77	0·9215
8	26·89	4·9	18·96	1·4559	36·96	0·9187
9	26·87	8·3	18·00	1·4541	28·2	0·9169
*A.	20·8	6·5	—	—	—	—
*B.	22·9	4·9	—	—	—	—

It can safely be assumed from these results that the usual presumptive limits for purity of the fat of butter do not apply to the fat of goats' butter; particularly is this the case with the Polenske value. This might perhaps be inferred if it is considered that the fatty acids obtained by the Polenske method may contain, in addition to lauric acid, lower members of the series which are also insoluble and which would be expected to be present from the following passage in an old edition of Allen's Commercial Organic Analysis: "The fat from the butter of ewes and goats' milk is very similar to that from cows' milk, but the esters of caproic and capric acids bear a larger proportion to the butyrin present than is the case of cows' butter."

The high Polenske figure obtainable with goats' butter is indicated in *König's Chemie der Menschlichen Nahrungs- und Genussmittel* (4th Edition *Nachtrag Zu Band*, 1, 1919, pp. 479–481), where the results of about 40 analyses of Danish, German and Russian samples are recorded. The Reichert-Wollny figures of these samples varied from 17 to 29, and the Polenske values from 4·15 (one sample 3·15) to 9·8. Two results recorded by Trimen for Egyptian goats' butter (ANALYST, 1913, **38**, 242) are included in the preceding table for the purpose of ready comparison with our results.

It is not intended at this stage to discuss these figures in detail, beyond drawing attention to the inference an analyst might at present attach to the Reichert-Wollny, Polenske and Kirschner values if he received similar samples described as butter.

The formula given by Richmond (*Dairy Chemistry*, 3rd Edition), to express the usual relationship between the Reichert-Wollny and Polenske figures is

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* Analyses by Trimen of Egyptian goats' butter.

$$\text{R.W.} \times 0{\cdot}033 - 0{\cdot}6155 = \log_{10}(\text{P} - 0{\cdot}48).$$

From this is deduced a formula by means of which the maximum allowable Polenske value may be calculated, *viz.* $\text{R.W.} \times 0{\cdot}033 - 0{\cdot}6155 = \log_{10}(\text{P} - 1)$.

Our Polenske values for goats' butter are compared with the maximum allowable for the various Reichert-Wollny values in the following table.

Reichert-Wollny value.	Polenske values.						
	Mean.	Maximum allowable.	Found in goats' butter.				
28	2·6	3·1	8·65	7·0	7·85	8·3	4·9
27	2·4	2·9					
26	2·3	2·8	8·7	7·6			
25	2·1	2·6	7·5	5·3			
24	2·0	2·5					
23	1·9	2·4					

Richmond further states that "should the Polenske figure exceed the maximum given in the table, the amount of coconut oil present may be deduced from the formula $\text{C} = \frac{\text{P} - \text{P}'}{14{\cdot}4} \times 100$."

where C = percentage of coconut oil; P = Polenske figure; P′ = Mean Polenske figure from the table equal to the Reichert-Wollny No. found + half the Polenske figure.

"Should palm-kernel oil be known to be present or detected by the test of Burnett and Revis, the figure 8·5 may be substituted for 14·4."

If the first formula were to be applied to our results, it might, erroneously, be inferred that our samples of genuine goats' butter contained the following percentages of coconut oil.

Sample ref.	1	2	3	4	5	6	7	8	9
Coconut oil, per cent.	12·64	11·46	9·51	7·64	14·24	4·17	10·42	0·35	10·78

The relationship between the Kirschner and Polenske values for cows' butter is expressed by Richmond as $\text{P} = (\text{K} - 14) \times {\cdot}26$, and he states that "it may be safely assumed that if the Polenske figure is higher than $(\text{K} - 10) \times {\cdot}26$, the presence of coconut or palm-kernel oil is established." That this formula could not apply to the samples of goats' butter we have analysed is shown.

Sample Ref.	Polenske value.	$(\text{K} - 10 \times {\cdot}26)$.
3	7·85	1·77
4	7·00	2·23
5	8·70	1·87
6	5·30	1·85
7	7·6	2·09
8	4·9	2·33
9	8·3	2·08

In every case the Polenske value is higher than that given by the formula, from which the same erroneous conclusion might be drawn, namely, that the samples contained a high percentage of coconut oil. The iodine values and refractive indices of the fat appear to correspond fairly well with those given by cows' butter, but the specific gravities appear to be distinctly higher and more variable. Determinations on many more samples would, however, be necessary before this could be asserted as being universally true. We had no information as to the diet or stage in the lactation period of the animals at the time our samples were taken, but we propose to study their effects on the composition of goats' butter in a further paper.

In conclusion, we would suggest that evidence has been produced to show (I) That the present legal interpretation of the words "Milk" and "Butter" should be more clearly defined.

(2) That the usually accepted standards do not hold good for goats' butter, and that an analyst receiving samples of this product designated as "butter," would almost certainly certify them as adulterated.

(3) That it is necessary to enforce that the prefix "goats' " be applied to the milk and butter obtained from goats.

We wish to acknowledge the help given by Mr. R. P. Hawkins, who carried out some of the specific gravity determinations, also assistance given by Mr. T. W. Palmer, Hon. Secretary of the British Goat Society, and those members of that Society who, at his request, kindly supplied the samples.

Chemical Department,
East Anglian Institute of Agriculture,
Chelmsford.

Discussion.

Mr. G. Rudd Thompson said that he had gained the impression that the use of artificial feeding stuffs, in which coconut might or might not appear, was indicated by the authors' results. The authors had stated that nothing at all was known of the diet of the goats, but he thought investigation would show the existence of compound feeding cakes for the use of goats. Milk produced by goats kept under hygienic conditions was known to be greatly on the increase, so that there was obviously no reason why goats' butter and milk should not be put upon the market, and anyone taking a case into Court would find it very difficult to prove that it was not "butter" or "milk," according to official standards, had the goat been in the vicinity of a coconut cake.

Mr. E. R. Bolton said that the Bombay Adulteration of Ghee Act defined "ghee" as made from the butter fat of the cow, buffalo, goat, or sheep; he, therefore, thought that the authors' figures for goats' butter would prove of interest to Indian chemists.

With regard to the question of substituting goats' butter for cows' butter, he thought that if a buyer asked for *butter* he would clearly expect to receive cows' butter. The speaker hoped that the authors would give any information they had about the diet of the animals, in view of the somewhat extraordinary figures, and asked if they had applied the baryta value of Avé-Lallement—a test which had proved useful for sorting purposes, giving a "minus" value for butter fat and a "plus" value for every other fat.

Mr. C. L. Claremont said that he thought that a cow would have to eat a very heavy ration of coconut cake for the analysis of the butter to show a figure like that mentioned by the authors for goats' butter.

Mr. C. A. Mitchell enquired whether the authors had done any work on determining the composition of the fat; if so, he would like to hear what proportion of stearic acid was present and what were the unsaturated acids.

Mr. G. D. Elsdon said the definition of "butter" was "the substance usually known as butter"; goats' butter was not usually known as butter, and therefore to supply goats' butter as butter would be adulteration. With regard to the constituents of the fat, would it not be advisable to find out the composition of cows' butter with some degree of accuracy before attempting that of goats' butter?

Mr. A. C. Barnes said the subject of the authors' paper was of considerable interest outside England. In West Africa there were no cows, and those fortunate enough to get milk got goats' milk; it was possible that ultimately there would be a large sale for both milk and butter from goats. As it was in these countries that the palm fruit was grown, the chances of adulteration of an animal fat by the natives were enormous, so that the authors' determinations were of great interest to people overseas. In his opinion, the authors should be encouraged to continue their investigations, and he suggested their obtaining samples of milk and butter from goats abroad.

Mr. A. More said that sheep' milk fat also gave higher Polenske figures than cows' milk fat, and recently he had examined the fat of a Roquefort cheese, made from sheep's milk, which had a Polenske value of 4·6, and a Reichert-Wollny figure of 27·5.

In reply to the discussion, the authors state that, although the full diets of the goats were unknown, they can assert that no coconut, palm-kernel or compound cake was used as far as eight samples of the butter were concerned.

In their opinion the high Polenske figure is natural to goats' butter. Their view is supported by the previous analyses quoted and by the fact that their samples were drawn from different counties. Feeding of cows on coconut cake has, however, been shown by Cranfield to influence the Polenske figure for the resulting butter, but the effect is comparatively slight.

The chief point in the legal position appears to have been missed by Mr. Elsdon—namely, that the definition of butter depends on the definition of milk. The authors agree that a buyer asking for butter might well expect to receive cows' butter, but are of opinion, that, as the law stands at present, he would have no redress if he received goats' butter.

No Avé-Lallement baryta values or proportions of constituent acids were determined, but it is hoped to make these determinations at a future date.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

DHOLL MIXED WITH DYED TALC.

"Dholl" is a dried lentil, of which five different types are imported into Natal from India. Recently the Indian Customs Authorities informed the Health

Authorities in this town (Durban) that a consignment of dholl had been sent here which was believed to be coloured with red lead. This material was traced to local Indian stores (we have a large Indian community here), and samples were purchased. No lead could be found in the dholl, but the samples contained from 6 to 22 per cent. of talc coloured with an eosine dye.

Apparently there is no question of adulteration, as the retailers state that certain castes will not purchase dholl unless it is so treated. I should be glad to have any information to explain this peculiar custom.

ALFRED A. KLOOT.

THE STEARIC ACID CONTENT OF BUTTER FAT.

THE method of estimating stearic acid in fats by crystallising the mixed fatty acids from alcohol already saturated at 0° C. with pure stearic acid was first described in a paper read before the Society by Hehner and myself (ANALYST, 1896, **21**, 316). In one of the sections in that paper we referred to the behaviour of butter fatty acids in that test, and mentioned that, whilst in many cases little, if any, deposit was obtained from them, in other cases some tendency to supersaturation was noticed.

As reference was made to this question in the discussion on a paper at the October meeting, I have thought it might be of value to place on record the results which, since that time, I have obtained with various samples of butter fat. Supersaturation may be prevented by adding a weighed excess of pure stearic acid to the mixed fatty acids as was suggested by Holland Reed and Buckley (*J. Agric. Res.*, 1916, **6**, 105; ANALYST, 1916, **41**, 209), but in my experience supersaturation only takes place, in the case of butter fatty acids, when a relatively small amount of stearic acid is present. Supersaturation may also be ended by shaking the flask vigorously after 12 hours and then leaving it for a further 12 hours in the ice-water. Referring to my experimental note-books, I find that the following results (*inter alia*) were obtained with samples of butter fat, which I had every reason to believe to be genuine:

	Reichert-Meissl value.	Stearic acid. Per Cent.		Reichert-Meissl value.	Stearic acid. Per Cent.
I.	23·1	15·7	XIII.	29·7	1·5
II.	—	nil	XIV. (Dutch)	18·6	21·7
III.	—	10·9	XVII. ,,	20·3	17·9
IV.	—	nil	XVIII. ,,	21·4	15·5
V.	—	nil	XIX.	—	nil
VI.	—	3·8	XX.	—	0·7
VII.	—	nil	XXI.	22·4	1·2
VIII.	28·8	(*a*) 4·1; (*b*) 4·3	XXII.	22·6	15·0
IX.	32·4	14·2	XXIII.	25·7	16·0
X.	30·8	15·2	XXIV. (Australian)	31·6	18·1
XI.	—	nil			
XII.	—	0·8			

In each case the deposit had the appearance of stearic acid crystals and melted between 68° and 69° C. In experiments in which weighed quantities of pure stearic acid were added to butter fatty acids the deposits contained these added amounts. Hence there does not appear to be any constituent in butter fat (analogous to that in Japan wax) which interferes with the crystallisation of stearic acid.

From these results it can hardly be doubted that the stearic acid content of butter fat may range from practically nothing to 22 per cent., at least.

It has been shown that such wide variations do occur in the body fat of different animals of the same species, and so it need not excite surprise that they should also occur in the milk fat. There is quite sufficient of the lower fatty acids in butter fat to form, with the stearic acid and oleic acid, mixed glycerides of relatively low melting point. C. AINSWORTH MITCHELL.

NOTES ON THE TREATMENT OF PARCHMENTS DAMAGED BY FIRE.

DURING the current year, in two separate instances, we have been asked to deal with legal documents—parchments—which had been contained in a safe during an outbreak of fire. The safe became involved, but showed a reasonable amount of resistance. The great heat, however, had baked the parchments to compact matter of much reduced bulk, and nearly as brittle as biscuit; they also had a rather pronounced burned and scorched appearance. It became necessary to refer to the subject matter of the documents, and the question was how to deal with them. On the first essay they were carefully dipped in warm water for some minutes, then put on cloths and left for some hours. It was found possible to open them out to some extent, but they were so unequally shrunken by the heat that the parts most baked were most contracted, although other portions opened out fairly well. It was then seen that the ink (the documents were of some age) was either faded or reduced by the water treatment, and those places were accordingly brushed over with a solution of gallic acid on a soft camel-hair brush. The documents were thus rendered legible, with the exception of a missing word or two which could be pretty well supplied from the context, and a copy required was made.

Then occurred a mischance with one of the documents which, after treatment with gallic acid, had been put between sheets of clean blotting paper and pressure gradually applied in a copying press. The moisture penetrated the layers of paper and brought away enough iron in solution from the press to stain the document, but fortunately it remained more or less legible, and the copy previously in use was available. It was found that directly the parchment began to dry again, the brittleness of the worst places returned with its accompanying risk of cracking or breaking; this was surmounted by brushing with a solution of about 1 part of glycerin to about 6 parts of distilled water, and thus producing a continuing dampness.

This treatment had a fair amount of success, but a much improved procedure was devised when a further parchment was sent to us some months later.

In this case a large beaker was taken and the bottom covered with cotton wool. The document, which in its shrunken state measured about 5½ in. by 2 in. by ¾ in. in thickness, was set upright on the wool, and from a flask provided with a safety tube steam was gently introduced into the beaker at the level of the cotton wool, which acted like a sponge as regards condensed water; the inlet of the delivery tube was at the side of a dial-glass and rested in the spout of the beaker. The operation went on for half an hour, and then the beaker and document in it were left at rest for a few hours. This treatment was quite successful; the ink had not run, and the document (although it would not properly flatten) could be opened without breaking and was manageable, especially after the glycerin treatment, which was applied earlier than in the former case. No gallic acid was used, nor was it necessary. Of course, there was not a very close resemblance to the original parchment, but it was distinctly legible, including the signature. In all cases the wax of the seals had melted in the first instance, and in one case the seal had been removed to the other side of the document, but any adhesion caused by the wax yielded to the high temperature, and the surfaces could be drawn apart. We were the more satisfied to get a pretty good result, as the case had been regarded as rather hopeless.

C. G. MATTHEWS.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

BOROUGH OF PORTSMOUTH.

REPORT OF THE PUBLIC ANALYST FOR THE YEAR 1923.

THE total number of samples examined during the year was 1336, of which 1202 were Food and Drug samples, and 46 were samples under the Milk and Dairies Amendment Act. Of the Food and Drug samples, 66 were found to be adulterated, and 9 of inferior quality. Twenty-eight of the 588 samples of milk, examined were adulterated and 5 inferior.

THE MILK AND DAIRIES (AMENDMENT) ACT (1923).—Six samples of "certified milk" were examined, and one was rejected. This sample contained 7300 bacteria per c.c. (standard not more than 30,000), but *B. coli* was detected in 0·1 c.c.

It is reasonable to suppose that in framing the Act, milk of at least average quality was pre-supposed, whereas all the samples of "certified milk" examined have been below the average in fat content (3·0 to 3·3 per cent.). This milk has been sold at an average retail price of 1s. 2½d. per quart.

Grade A Milk.—This milk is to be produced and treated under such conditions that a sample taken at any time between production and delivery to the customer shall not contain "more than 200,000 bacteria per c.c., and *B. coli* shall be absent from 0·01 c.c. of milk." The average retail price of this milk during the year has been 9d. per quart.

For comparison purposes the results of 20 of the many examinations of *ordinary* milk as supplied to hospitals and other local institutions are given. The numbers of bacteria per c.c. ranged from 1720 to 32,100. Ten of the samples gave negative results in the test for *B. coli* in 0·01 c.c. of milk.

These results indicate that a standard of 200,000 bacteria per c.c. is vastly in excess of what a milk produced under normally sanitary conditions should contain.

It is suggested that the present specifications for hospitals and other municipal institutions should be altered, and that in future only "Grade A" milk be accepted.

PRESERVATIVES.—In three of eleven samples of cream sold as preserved cream no boric acid was present, and the remainder contained from 0·03 to 0·16 per cent.

Boron preservative was found in 55 samples of butter (41 per cent. of those examined), but in no case in greater proportion than 0·29 per cent.

REGINALD P. PAGE.

COUNTY BOROUGH OF SALFORD.

ANNUAL REPORT OF THE BOROUGH ANALYST FOR 1923.

DURING the year 1591 samples were submitted for analysis, of which 1388 were taken under the Sale of Food and Drugs Acts, and 203 were submitted by various Corporation departments. Of the Food and Drugs samples 96 (6·9 per cent.) were returned as adulterated.

MILK.—Of the 779 samples examined, 42 (5·4 per cent.) were returned as adulterated.

Two samples contained 0·01 per cent. of potassium nitrate, and 2 contained 1 part of potassium nitrate in 50,000. Proceedings were taken against the vendor of these samples, and he was fined £3 for deficiency in fat. The Magistrate remarked that he would not, on that occasion, impose a penalty for the presence of the potassium nitrate, but hoped that the case would serve as a warning to others in future.

Dirt in Milk.—Since the year 1915, 2765 average samples of milk coming into the borough have been examined. Of these, 946 have been entirely free from sediment, 1208 contained between 0 and 1 part per 100,000, and 218 have contained between 1 and 2 parts per 100,000. Hence, since 81 per cent. of the samples have contained not more than 1 part per 100,000, it would appear that that would be a fair standard, and that milk containing 2 or more parts per 100,000 should be classified as unsatisfactory. Efforts are being made, by administrative methods, to improve the present condition of the milk supply in this respect, before taking more drastic steps.

BUTTER AND MARGARINE.—Of 53 samples of butter examined, 9 were preserved with boron preservative (=0·3 per cent. of boric acid).

Of 43 samples of margarine none was adulterated. Seventeen were tested for boron preservative, which was present in every case. In nine it amounted to 0·4 per cent. as boric acid.

Legal proceedings were instituted in several cases for the sale of margarine as "butter mixture," one of the samples containing only 0·5 per cent. of butter. Fines were inflicted in 2 cases. (*Cf.* ANALYST, 1923, **48**, 489; 1924, 85).

CHEESE.—Fifteen samples of cheese contained pure milk fat in the proportions of 21·5 to 47·0 per cent.

Eight samples of cream cheese were examined, and, of these, 4 (containing between 5 and 6 per cent. of milk fat) were returned as adulterated. (*Cf.* ANALYST, 1924, 264).

A few cheeses were sold as "Bondon Cheese," and of these samples, some contained 38 per cent. of fat, whilst others contained only 5 per cent. A special report was made on the subject (see ANALYST, 1924, 267).

BEEF AND MALT WINE.—Proceedings were taken against the vendors of beef and malt wine deficient in both meat extract and malt extract, and the maximum fine, with 20 guineas costs, was inflicted. The analyses were accepted, the amounts of meat and malt extracts actually added being about half the maximum amount mentioned in the certificate ("not more than 0·2 per cent. of a mixture of equal parts of meat and malt extracts"). (ANALYST, 1923, **48**, 326; 1924, 210.)

SYRUP TREACLE.—Of 32 samples of golden syrup examined, 22 were found to be genuine golden syrup derived from cane or beet sugar products, but 10 samples (from 3 manufacturers) contained a large percentage of glucose syrup. Proceedings were instituted in two cases, but both were dismissed, one on the ground that the label (though misleading) afforded some sort of protection, and the other because of evidence that the term "syrup treacle" was not a term usually known in the trade. In the Salford district, however, ample evidence can be found that the term was at one time well known. (*Cf.* ANALYST, 1923, **48**, 599, 600.)

JAM.—Eighteen samples from 13 vendors, out of 26 examined, contained from 8 to 20 per cent. of glucose syrup, and 1 contained 35 per cent. In the case of marmalade it has been held by the High Court (ANALYST, 1901, **26**, 329), that there was no evidence to show that a sample which contained 13 per cent. of glucose was not marmalade. This ruling would probably be followed in the case of jam, and those samples, therefore, which contained not more than 15 per cent. of glucose syrup and were not labelled in any objectionable way were passed as genuine.

Sixteen samples, from 11 manufacturers, were labelled in such way as to lead the purchaser to suppose that the jams were free from glucose syrup. The manufacturers were approached in the matter, and agreed either to drop the use of glucose syrup or to modify their labels, by omitting all references to sugar in the case of jams containing about 15 per cent. of glucose syrup, and by adding the words "scientifically preserved" when larger proportions of glucose were present.

ARSENIC IN FOOD WRAPPERS.—Nine of 51 samples examined contained an appreciable quantity of arsenic derived from the printing inks (*cf.* ANALYST, 1924, 336).

CANNED GOODS.—Of 35 samples examined, 12 of canned fish and 8 of canned tomatoes, were fairly satisfactory, since they contained less than 2 grains of metallic tin per lb. Fifteen samples of canned bristlings were reported to be unsatisfactory, as they contained from 2·7 to 6·7 grains of metallic tin per lb. The owners agreed to withdraw them from sale and destroy them.

G. D. ELSDON.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

CHOCOLATE.

ON September 3, a grocer was summoned at Stanley (Co. Durham) for selling as "chocolate" an article not of the nature, substance and quality demanded.

Mr. C. J. H. Stock, Public Analyst, gave evidence to the effect that the sample of "chocolate animals" in question was a sweetmeat not of the composition of chocolate. It consisted of a mixture of chocolate with gelatin, maize starch and ferric oxide, which last substance, in the proportion in which it was present, was not a constituent of genuine chocolate.

For the defence it was contended that the sweet was described by the manufacturers as "Chocolate Mixed Brown Animals," and that this was intended as a description of the colour. The article was not sold as chocolate.

A fine of £2 and costs was imposed.

ARSENIC IN BORAX.

On September 23rd a firm of druggists was summoned at the Mansion House for having sold as purified borax a sample of borax containing 120 parts of arsenic per million.

Mr. Earle, prosecuting for the Corporation of London, said that, as borax was taken internally as a medicine, it was of the greatest importance that it should be of the highest degree of purity, and that the standard of the British Pharmacopœia (in the case of this drug 5 parts of arsenic per million) should be complied with.

Mr. H. Glyn-Jones, for the defence, said that in this case a good commercial sample of borax had been supplied instead of the B.P. article. Borax might occasionally be used as a medicine; it was sometimes used in mouth-washes; more often in toilet preparations, but mainly for industrial purposes, such as laundering. He submitted that it was inconceivable that borax containing not more than 120 parts of arsenic per million could do harm. One would have to take more than 20 times the average B.P. dose of borax in order to get an average dose of arsenic. To get a really poisonous dose of arsenic about a pound and a half of this borax would have to be swallowed.

The Lord Mayor, on being assured that the consignment of borax had been destroyed, said that he was prepared to deal leniently with the case, and imposed a fine of 40s. with 2 guineas costs.

ADULTERATION OF SHELL EGGS.

The following case is typical of ten cases reported by the U.S. Department of Agriculture:*—On July 5th, 1923, the United States Attorney for the District of Nebraska, acting upon a report by the Secretary of Agriculture, filed in the District Court of the United States for the said district an information against a firm of traders, of Danbury, Nebr., alleging shipment by said defendants, in violation of the Food and Drugs Act, on or about August 9, 1922, from the State of Nebraska into the State of Colorado, of a quantity of shell eggs which were adulterated.

Examination by the Bureau of Chemistry of 1080 eggs from the consignment showed that 204 or 18·88 per cent. of those examined, were inedible eggs, consisting of "black rots, mixed or white rots, mouldy eggs, spot rots, and blood rings." (*Cf.* Analyst, 1924, 279.)

Adulteration of the article was alleged in the information for the reason that it consisted in part of a filthy and decomposed and putrid animal substance.

On March 3, 1924, the defendants entered a plea of guilty to the information, and the Court imposed a fine of $25.

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* Service and Regulatory Announcements, Bureau of Chemistry. Supplement. N.J., 12201–12250. Aug. 1924. (No. 12212, *United States* v. *W. T. Harris*. F. & D., No. 17419, I.S., No. 7583–v.)

Ministry of Health.

FUR DERMATITIS.*

In this enquiry into the occurrence of dermatitis attributed to the wearing of fur collars the subject has been investigated from all sides. Information as to dyeing process has been obtained from the fur trade associations and from private individuals, and the co-operation of dermatologists, medical officers of health and chemists has been enlisted.

Although cases of dermatitis caused by the wearing of dyed furs have been recorded by dermatologists during the last 20 years, there is no history of any aggregation of cases similar to the "epidemic" of the winter season of 1923–1924, when the total number of cases up to July, 1923 (when complaints ceased), probably ran into hundreds. Only a relatively small number of fur-wearing women and girls were affected, so that personal susceptibility was responsible to a certain extent. In most cases there is a latent interval of 2 to 3 weeks, but sometimes the symptoms appear within a few hours of contact with the fur.

The furs which have been proved to be the cause of the complaints are rabbit skins dyed to resemble beaver; they are exclusively produced and dyed in foreign countries, notably Belgium, France, Germany, and the United States.

Defective or careless technique in the use of potentially dangerous dyes is, in all probability, the cause of the trouble. (*cf.* ANALYST, 1923, **48**, 282, 283, 284).

BACTERIOSCOPIC EXAMINATION.—The report contains in an appendix a report by Dr. Eyre of Guy's Hospital on the bacterioscopic examination of a fur collar that had caused dermatitis. The hairs were normal in character, no bacteria or moulds could be demonstrated microscopically, and cultures from the hair and skin failed to yield any micro-organisms which could be associated with dermatitis.

CHEMICAL EXAMINATION.—Appendix 4 contains a report by Professor Perkin on the chemical examination of four samples of suspected furs. The conclusion drawn is that there can be no doubt that the trouble has arisen in connection with the so-called oxidation colours, which are developed on the fibre from colourless substances, probably all of which are of a poisonous or irritating character.

Whereas in (A) no compound of the nature of *p*-phenylene-diamine could be detected, in (B) *p*-phenylene-diamine was actually present; (C), on the other hand, contained traces of a readily crystalline soluble basic aromatic substance, the exact chemical identification of which was not possible, but which, it is reasonably suspected, was allied to *p*-phenylene-diamine itself. Finally, (D) in this respect resembled (A).

All the furs contained a substance or substances, in moderate amount, soluble in alcohol, and from these solutions brown semi-crystalline products were obtained possessing a remarkable similarity to Bandrowski's base, the well-known oxidation product of *p*-phenylene-diamine, which is readily obtained by passing air through an ammoniacal solution of *p*-phenylene-diamine, and is also, without doubt, an oxidising agent.

The colouring matter present in these furs was by no means of a homogeneous character, for although much was insoluble in the usual solvents, some portions were, however, soluble in water, in boiling alcohol, and in cold dilute hydrochloric acid respectively.

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* Reports on Public Health and Medical Subjects. No. 27. By Allan C. Parsons, M.R.C.S. H.M. Stationery Office, 1924. Price 9d. net.

The conclusions with regard to these points are given with reserve, as an authentically innocuous dyed fur for purposes of comparison could not be obtained. On the other hand, this lack of homogeneity of colouring matter is likely to have arisen from a faulty application of the dyeing process—in other words, an incomplete oxidation of the developer to the finished dyestuff. Whereas in the case of the furs (A) and (D), *p*-phenylene-diamine or an analogous compound was absent, and these furs had undoubtedly produced dermatitis, it is thus probable that the irritant was a product representing an early stage of colour development that is of the nature of Bandrowski's base. Again, although in fur (B) *p*-phenylene-diamine was found, and in fur (C) there was a crystalline substance of an apparently similar character, there was also present in each immature colouring matter, closely similar to Bandrowski's compound. Finally, all the furs had a faintly acid reaction and showed no sign of the application of mordants.

No information is available as to whether in high-class fur-dyeing the mordanting operation is considered to be essential for a complete development and fixation of the dye. Such an adjunct to the dyeing operation is, however, evidently very usual, and is described in the majority of fur-dyeing recipes. An after-treatment of this character may, on the other hand, be most important. Thus, according to Austin (*Principles and Practice of Fur Dressing and Fur Dyeing*, p. 168), "Some shades, especially black, have a tendency to rub off slightly. To overcome this the dyed furs are treated with a cold solution of one part of copper sulphate in 1000 parts of water, for 3 to 4 hours," and (p. 160), "It was this superficial deposition of minute crystals of the dye, or the only partially oxidised intermediate . . . which was frequently injurious to the health."

Finally, the following note of W. F. Castle (ANALYST, 1923, **48**, 284), is interesting:—"The furs seem to be innocuous unless the wearer has a greasy skin, when the colouring agent appears to dissolve in the fatty acids."

BIBLIOGRAPHY.—The Report concludes with a bibliography containing 28 references, dating from 1902 to 1924.

FINAL REPORT OF THE DEPARTMENTAL COMMITTEE ON THE USE OF PRESERVATIVES AND COLOURING MATTERS IN FOOD.*

FOLLOWING a summary of the regulations adopted by other countries in the matter of preservatives and colouring matters in food, the effect of various preservatives on the consumer and their relative harmfulness is discussed, and, from a consideration of the evidence, the conclusion is drawn that preservatives may be classified in three groups according to their relative degrees of undesirability, *viz.*: Group I., Formaldehyde and its derivatives; hydrofluoric acid and its derivatives. Group II., Boron preservatives; salicylic acid and its salts. Group III., Benzoic acid, sulphurous acid and their salts.

The existing practice in connection with various foods is next described, and after a general consideration of the evidence of medical men and traders, each group of foods is dealt with in turn.

MILK.—The addition of any preservative in milk is now prohibited by the Milk and Cream Regulations, but antiseptic agents are still advertised and sold for sterilising dairy utensils and milk bottles. To prevent possible contamination of milk from this source, the Committee are of opinion that only steam or other suitable source of heat should be permitted for this purpose.

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* H.M. Stationery Office. Pamphlet 84. 1924. Price 1s. 6d. net

CREAM.—Given appropriate care to preparation and the use of suitable methods of transport, storage and distribution, the Committee see no sufficient reason why the use of preservatives in cream should not be prohibited after a sufficient period of notice to enable the trade to make suitable adjustments.

BUTTER.—The conclusion reached after a consideration of all the evidence is that a period of two years would be sufficient to allow such adjustments of method to be made as would enable all butter to be produced and sold without preservatives. The Australian and New Zealand trade, and possibly that of the Argentine, seemed to be the only cases in which any difficulty was to be apprehended. From both the former countries a considerable quantity of butter is already being sent without preservatives. The requirement of compulsory pasteurisation which has been recently imposed in Australia will, it is believed, help to improve the keeping quality of the butter, and the Committee have little doubt that such a regulation as we suggest would stimulate improvements in other directions.

MARGARINE.—The Committee are of opinion that well-made margarine keeps as well as, or even better than butter, and therefore recommend that the addition of preservatives should be prohibited.

SAUSAGES.—As at present there is a lack of suitable transport and storage accommodation, the conclusion has been reached that, in existing circumstances, some preservative should be permissible (*vide infra*).

BACON AND HAM.—The Committee consider that difficulties of keeping these foods can be satisfactorily met by the provision or extension of suitable storage accommodation on ship and on shore, and therefore advise that, after due notice, the use of preservatives in the preparation, storage or distribution of bacon and ham should be prohibited.

BRAWN, POTTED MEAT AND POTTED FISH.—It is recommended that the use of all preservatives be prohibited in these products, including potted shrimps.

MEAT.—It is recommended that the use of all preservatives should be prohibited, no exception being made in favour of the Bullot process of exposing the freshly-killed carcase to sulphurous fumes derived from a special mixture. After this treatment as much as 2 grains of sulphur dioxide per lb. could be detected throughout the meat.

SUMMARY OF CONCLUSIONS AND RECOMMENDATIONS.

1. Preservatives should be prohibited in all articles of food and drink offered or exposed for sale whether manufactured in this country or imported, except that

(A) Sulphur dioxide only should be permitted

(i) in sausages in amounts not exceeding 3 grains per pound; (ii) in jam in amounts not exceeding 0·3 grain per pound; (iii) in dried fruit in amounts not exceeding 7 grains per pound; (iv) in preserved (but not dried) whole fruit or fruit pulp in amounts not exceeding 5 grains per pound; (v) in beer and cider, whether in bottle or in cask, in amounts not exceeding 5 grains per gallon; (vi) in alcoholic wines, non-alcoholic wines and cordials, and fruit juices, sweetened and unsweetened, in amounts not exceeding 3 grains per pint.

(B) Benzoic acid only should be permitted

(i) in coffee extract in amounts not exceeding 3 grains per pound; (ii) in non-alcoholic wines and cordials, and sweetened and unsweetened fruit juices (as an alternative to sulphur dioxide) in amounts not exceeding 5 grains per pint; (iii) in sweetened mineral waters and in brewed ginger beer in amounts not exceeding 1 grain per pint.

The method of estimating the foregoing preservatives should be prescribed by the Minister of Health.

2. The sale of any preparation as a food preservative or for use in such circumstances that it may be introduced into food should be declared illegal unless such preparation

(*a*) bears a description clearly indicating its composition and strength; (*b*) is free from impurities and, in particular, contains not more than 1/100th part of 1 grain of arsenic per pound or more than 1/7th part of 1 grain of lead per pound.

3. The use of preservatives, so far as they are permitted, should be upon the condition that the nature and quantity of the preservative present in the article of food should be declared in a manner prescribed by the Minister of Health. In cases in which the declaration of the preservative might be difficult to enforce or might result in undue harm to the industry without compensating advangage to the consumer, exception to this requirement might be made, but the exception should in no circumstances apply to sausages.

4. The employment of a copper salt to colour or preserve the colour of peas and other vegetables should be prohibited.

5. A schedule should be issued by the Minister of Health, after such enquiry as is necessary, of colouring matters the use of which may be considered non-injurious to health, such schedule to be subject to amendment or extension from time to time as occasion may require. Within a suitable period after the publication of this list the use of any other colouring matter should be prohibited, unless and until it shall have been approved by the Minister.

6. Before the prohibition of preservatives or of colouring matters in food (including the use of copper salts for the so-called greening of vegetables) is enforced, a period of grace should be allowed sufficient to enable manufacturers and importers to adjust their methods and processes and to allow stocks to be cleared.

7. Improved methods in the storage and transport of food by rail, road and water, especially as regards the use of refrigeration and cool air storage, are urgently required.

8. It should be provided by law that any Regulations or Statute prohibiting or limiting the use of preservatives and colouring matters should bind the courts in proceedings taken under the Sale of Food and Drugs Acts in respect of their use.

9. An amendment of the law is required to render more expeditious the prosecution of a person actually responsible for offences under these Acts, where a warranty defence is pleaded.

10. Further powers of control by registration, licensing or inspection should be given to Local Authorities in relation to all places concerned in the production, sale, storage, and distribution of food.

Report of the Government Chemist upon the Work of the Government Laboratory.

FOR THE YEAR ENDING MARCH 31ST, 1924.*

THE chemical work of the following Departments is carried out wholly or in part at the Government Laboratories—Admiralty, Ministry of Agriculture and Fisheries, Air Ministry, Colonial Office, Crown Agents for the Colonies, Board of Inland Revenue, Ministry of Pensions, Post Office, Department of Scientific and Industrial Research, Home Office, Board of Trade, War Office, Office of Woods and Forests, Privy Council, and Office of Works. The total number of samples examined during the year, including those dealt with at the chemical stations, was 390,421, an increase of 46,968 over the preceding year. Marked increase is shown in the number of wine, sugar and tea samples, and of wort, exported beer, and spirituous preparations. The number of imported beer samples rose from 1361 to 15,283, those of exported tobacco and snuff doubled, these latter being largely due to the examination necessitated by the establishment of the Irish Free State.

IMPORTED DAIRY PRODUCE AND MARGARINE.—*Butter.*—Of the 867 samples examined, 10 exceeded the 16 per cent. water limit, and 3 "hard butters" were palm-kernel preparations.

Margarines.—Five out of 455 samples exceeded the water limit.

Cheese.—The fat varied in the 138 samples examined from 8·6 to 38·1 per cent., or 21·0 to 56·9 per cent. on the dry matter.

Cream.—The tinned creams contained from 23 to 54 per cent. of fat, and the preserved creams from 51 to 67 per cent.

Milk.—Two out of 6 samples of tinned sterilised milk taken from specimen consignments sent with the object of getting a footing in this country had been deprived of about a quarter of their fat, and another sample was incorrectly labelled.

Condensed Milk.—One hundred and thirty six samples were examined under the old regulations, and 263 under the new regulations which require that the milk should conform to the following standards:

Whole milk, sweetened or unsweetened,	31 per cent. of milk solids, including 9 per cent. fat.
Skimmed milk, sweetened,	26 per cent. milk solids.
Skimmed milk, unsweetened,	20 per cent. milk solids.

Of these, 12 samples were deficient in fat, and 3 of these in milk solids as well; 10 overstated the contents of milk represented, and in 15 the fluid produced by the stated dilution was below the prescribed standard. Labelling errors appeared in 17 samples.

SHEEP DIPS.—Samples of dips are examined for suitability of formula and conformation to formula, and baths for strength. Thirty-six out of 134 samples were reported as defective.

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* H.M. Stationery Office, Kingsway, W.C. Price 1s. 6d. net.

Water and Pollution of Rivers.—From the point of view of fish life, 18 samples of river water and effluents were examined; also 32 samples of domestic water, and 12 of road dressings for harmful substances.

Sea Water.—The salinity of 5757 samples and the amount of dissolved oxygen in 131 samples of sea water was determined, chiefly in connection with the influence of the salt concentration upon fish life.

Fertilisers and Feeding Stuffs Act.—During the year 10 fertilisers and 13 feeding stuffs were reported upon. Two samples of basic slag contained admixed mineral phosphates, 1 sample was deficient in citric-soluble phosphates, and in 3 the guarantee of citric-soluble phosphates was greatly understated. A sample of bone siftings was deficient in phosphates and contained over 10 per cent. of sand. Of the feeding stuffs, the ground oats were adulterated with from 20 to 80 per cent. of barley meal and oat-shudes, a sample of sharps contained 4 per cent. of rice husks, a sample of pea meal 40 per cent. of wheat, rye and dari, feed and cotton cakes were deficient in oil and albuminoids, and 3 samples of fish meal contained excess of oil. The statements of oil and albuminoid content given with samples of fish, meat and bone meals were too low to represent the composition of any such articles on the market. In only 1 case was there disagreement with the Agricultural Analyst who examined the samples in the first instance.

Merchandise Marks Act.—Out of a total of 21 samples examined, 1 sample of ground oats contained 15 per cent. of tapioca flour (conviction and fine), another 85 per cent. of barley and tapioca; 3 samples of pea meal contained 5·25 and 33 per cent. respectively, of maize and barley; 4 samples of barley meal had 25, 30, 40 and 50 per cent. respectively, of oat refuse and weed seeds, and a fish meal contained 10 per cent. of ground cereals and refuse. Two samples of copper sulphate contained 50 per cent. of iron sulphate, and a sample sold as cherry cider was found to be an artificial preparation.

Miscellaneous Articles.—Out of 24 samples of apples examined for evidence of insecticidal sprays, one showed 1/30th of a grain of lead, and arsenic equivalent to 1/50th of a grain per lb. A sample of milk powder contained 25 per cent. of vegetable fat.

Customs and Excise.—In connection with duty on beer, 47,733 samples were examined, and 385 samples of brewing materials were tested for arsenic, of which 90 slightly exceeded the limit of the Royal Commission of 1/100th of a grain of arsenious oxide per lb. for solids or per gallon for liquids.

Cocoa and Chocolate.—The estimation and detection of cocoa butter substitutes in chocolate has continued to receive attention and 3020 samples of chocolate confections were found to contain spirit.

Dangerous Drugs Act.—Out of 94 samples, 24 were found to contravene the Act. Two samples marked "morphiumpulver" were found to consist of lactose.

Dyestuffs.—Three hundred and fifty-eight samples of imported colours, lakes, and other goods were examined.

Matches.—None of the 153 samples contained white phosphorus.

Safeguarding of Industries Act.—Nearly 8000 samples of medicinal preparations, toilet articles and perfumes were examined, many of the analyses being of an intricate nature.

Spirits.—One sample of wood naphtha out of 787 was not approved for use in the preparation of methylated spirits, and 993 samples of special denaturants were examined. One additional denaturant has been recommended for mixing with methylated spirits. The strength of spirit or quantity of sugar in gin, brandy, etc., for export was overstated in 360 claims for drawback out of 2490, and the spirit strength in 276 cases out of 17,700 for medicinal spirits, tinctures, etc. Fourteen samples of fusel oil out of 50 contained more than 15 per cent. of proof spirit, and the proportion was allowed to be reduced as an alternative to paying duty.

Sugar, Glucose and Saccharin.—Although fixed rates of duty have been established for a large number of articles containing sugar, many substances have still to be tested on each importation, and 55,887 articles were examined for assessment or drawback, 1831 samples of glucose and 199 of saccharin, or articles containing saccharin, besides 160 articles for the presence of saccharin.

Table Water Duty.—Of the 100 cordials, syrups, etc., 4 contained between 2 and 5 per cent. of proof spirit, and all the non-alcoholic wines less than 2 per cent.

Tea.—Of 42,068 samples examined, 1024 were reported against, representing 4853 packages of tea; 394 on account of the presence of foreign substances, and 630 as unfit for human consumption; 1095 samples of denatured tea were examined.

Tobacco.—Moisture was estimated in 400 samples of imported and home grown tobacco, and in 9833 of manufactured tobacco, and oil in 1313 samples from stocks of manufacturers and retailers. The number of samples of tobacco exported on drawback was 16,662, as against 8262 last year, the increase being largely owing to export to the Irish Free State pending establishment there of branch factories by British manufacturers. This factor was also responsible for the increase in snuff samples from 271 to 1056. During the year 22,764 samples of tobacco stalks for drawback and 6964 for test as to uniformity of condition, together with 6294 samples of offal snuff, shorts and smalls, were examined. One sample of commercial snuff contained prohibited ingredients, as did 19 out of 128 samples of essential oils, whilst 1 of tobacco contained formaldehyde. Aluminium foil was found to be more satisfactory than lead foil for enclosing tobacco submitted for moisture tests. Experimental research into the composition of British-grown tobacco at various stages is being undertaken with regard to the quantities of nicotine and sugar present.

Wines.—In all, 92,319 samples were tested.

HOME OFFICE.—The usual examinations were continued and an explosion in an oxygen compressor was traced to the lubricant of soap water. Copper and lead soaps were formed and these split off their fatty acids owing to the local heating, and exploded in the air. A case of hydrogen arsenide poisoning was found to be due to dross from a metal foundry in bags becoming saturated with rain water.

DEPARTMENT OF SCIENTIFIC AND INDUSTRIAL RESEARCH.—A series of experiments was carried out on the recovery of formaldehyde by means of water. The results obtained on the investigation of the ammonium sulphate nitrate involved in the explosion at Oppau were described at a meeting of the Faraday Society on the 14th April, 1924.

BOARD OF TRADE.—The quantity of lime and lemon juice approved for the Mercantile Marine was 24,376 gallons.

CORPORATION OF TRINITY HOUSE.—The cause of sediments in oil storage tanks and the effect of certain grades of oil on lead pipes was enquired into.

OFFICE OF WORKS.—The examination of samples of materials purchased for the Public Services was continued, and also the enquiry into the decay of stone in town atmospheres. Chlorinated hydrocarbons used in charging certain types of fire extinguishers were found liable to cause corrosion of the metal, particularly in the presence of moisture.

SALE OF FOODS AND DRUGS ACT.—Thirty samples of food (including 29 of milk) and 5 samples of drugs were examined. There were four cases of disagreement with the results put forward by the prosecution; two samples of medicine alleged to contain excess of quinine sulphate were found deficient in that substance, 2 samples of milk alleged to be deficient in non-fatty solids were found to contain 8·50 and 8·54 per cent., respectively, whilst there was partial disagreement in the case of a sample of milk alleged to be deficient in fat and in non-fatty-solids, which was found to be deficient in fat but to contain 8·61 per cent. of non-fatty-solids.

D. G. H.

Methods for the Determination of Sulphur and Moisture in Pyrites Ores.

AS APPROVED BY THE SWEDISH GOVERNMENT TESTING INSTITUTE AND BY THE NORWEGIAN GOVERNMENT ANALYSIS COMMITTEE.

I. INTRODUCTION.

THE method here described contains detailed instructions for the preparation of the general sample for analysis after its arrival at the laboratory and for the method employed in the analysis. In regard to sampling, the following general information and hints, only, will be given.

In order that the chemical analysis shall give reliable values for the composition of a certain lot of pyrites ore, the samples analysed must be correct average samples (general samples) of the lot in question.

The sampling is usually carried out by taking from various portions of the lot in question a larger rough sample, which is then repeatedly ground, mixed and subdivided until a certain sample quantity, having the desired fineness, is obtained, from which eventually one or *simultaneously* a number of *identical* samples are taken for subsequent analysis at various laboratories. The samples taken for the determination of moisture are called *moisture samples*, and those taken for the determination of sulphur are called *quality samples*.

Moisture samples may be suitably taken after the general sample has been reduced to particles not exceeding 10 mm. in size. At this size of grain at least 2·5 kg. should be taken for each moisture sample. The samples are immediately packed as described below.

During the taking of the general sample and during its preparation for a moisture sample, care must be taken that the moisture contents remain unchanged.

Quality samples may suitably be taken after continued repeated subdivision and crushing of the general sample down to a size of 1 or 2 mm. Where the particles do not exceed 2 mm. in size, each quality sample taken should weigh not less than 1·2 kg., and when the largest particle has 1 mm. diameter, each quality sample should weigh at least 0·3 kg.

The samples are immediately hermetically enclosed (in, for instance, a glass bottle with a tight-fitting cork, or tin vessel closed by soldering) and given a label with the necessary information, of which a copy is laid on the top of the sample inside each packing and sealed with the seal of the sampler.

II. PREPARATION OF SAMPLE FOR ANALYSIS.

The *moisture sample* is subjected to no special preparation and is used in its original conditon for the determination of moisture (see below).

The *quality sample* is dried, if necessary, at normal room temperature (spread on a suitable surface) or else in a drying cupboard at a temperature not exceeding 40° C. The sample is then crushed and subdivided into two analysis samples with a maximum particle size of about 0·1 mm. according to the following scheme:

Weight of sample.	Crush to maximum particle.	
2·5 to 10 kg.	abt. 2 mm.	After crushing, the sample is mixed and halved repeatedly until a quantity of not less than 1·2 kg. is obtained.
0·3 to 2·5 kg.	abt. 1 mm.[*]	After crushing, mix and halve repeatedly until a quantity of not less than 0·3 kg. is obtained.
0·3 kg.	abt. 0·25 mm.	After crushing and mixing, 2 ultimate samples, each of at least 10 grms., are taken and ground in an agate mortar down to a size of about 0·1 mm.[†] The samples are kept after subdivision in closed weighing bottles.

Crushing down to 2 mm. and 1 mm. generally takes place in a rolling mill or on a steel plate with the use of a sledge-hammer. In the latter case great care must be taken that the crushing is done by careful blows with the hammer and not by grinding, as pyrites ore, on account of its hardness, will be contaminated with iron from the plate and the hammer. Crushing from 1 mm. down to 0·25 mm. is done in a small ball mill with steel balls (for instance, the model of the Government Testing Institute) or on a steel plate with a sledge-hammer as before.

The utility of special crushing tools should be separately investigated, for instance by comparative analysis of two identical samples, of which one has been crushed on a steel plate as described above.

III. THE ANALYSIS.

Determination of Moisture.

After careful mixing of the moisture sample (but without crushing) two samples of 0·1 to 1 kg., depending on the size of particles, are taken and dried in a drying cupboard at 105° C. until a constant weight is observed. As a result the average value of the two tests is given.

Determination of Sulphur.

Analyses are carried out on both the ultimate samples ground in the agate mortar according to one of the following two methods.

As certain ores after grinding rapidly increase in weight on account of oxidation, one should not wait more than 24 hours before weighing in the analysis, and for the same reason the subdivision[†] of the sample should not be carried too far.

METHOD A: SWEDISH-NORWEGIAN METHOD ACCORDING TO LUNGE.

1. Weigh in 0·5 grm. of the finely pulverised ore sample and transfer the weighed quantity to a glass beaker of 250 to 300 c.c. (low model).

2. Add 20 c.c. newly mixed nitric-hydrochloric acid (3 parts nitric sp. gr. 1·40 + 1 part hydrochloric sp. gr. 1·19) and let the beaker stand, with a watch glass cover, without warming or shaking, until the ore appears to be completely dissolved, preferably until the following day.

Nitric acid of a lower sp. gr. than 1·40 should not be used for this purpose. A somewhat stronger acid may, however, advantageously be employed. In the case of some ores a small quantity of sulphur separates during solution, but it dissolves completely when the sample is left at room temperature or during the succeeding heating.

3. Warm the sample on a boiling water bath for about ¾ hour. Remove the watch glass (do not swill) and evaporate to dryness on the water bath.

The beaker should not be sunk in the water bath. After about ¾ hour's heating there will be no appreciable traces of sulphuric acid on the watch glass, for which reason the acid mixture should not be unduly diluted by swilling the cover.

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[*] During crushing, the fine material may with advantage be sifted off repeatedly through a sieve with 1 mm. mesh, after which the remnant on the sieve is again crushed.

[†] If the sample is shaken through a sieve with 140 mesh per inch (0·12 mm. free opening) the remainder on the sieve should amount to 2 or 3 per cent. When grinding is carried too far the sample is considerably altered either by oxidation or by admixture of silica from the agate mortar.

4. Dissolve the residue in 5 c.c. diluted hydrochloric acid, sp. gr. 1·12, while heating with a cover over the beaker. Swill the glass cover and evaporate again to dryness on water bath.

In employing dilute hydrochloric acid, sp. gr. 1·12, no evanescence occurs with the attending spitting, as when conc. hydrochloric acid is used. Any nitric acid remaining in the residue is, however, completely driven off by evaporation with 5 c.c. dilute hydrochloric acid.

5. Moisten the evaporation residue with 2 c.c. hydrochloric acid, sp. gr. 1·19, and heat until all except the insoluble residue (gangue, silica, etc.) is dissolved. Swill the covering glass and dilute to about 100 c.c. with warm water. Filter off the insoluble residue and wash the same with about 50 c.c. warm water. Let the filtrate pass into a beaker of 250 c.c. capacity.

In adding hydrochloric acid a dropping-bottle may be used with advantage, the number of drops of hydrochloric acid per c.c. having been determined.

6. Precipitate in the warm solution the ferric hydroxide with 20 c.c. ammonia, sp. gr. 0·96 (or 15 c.c. sp. gr. 0·95), heat to 60 or 70° C. for 15 minutes, filter and wash the precipitate on a 10 cm. filter. Filtrate and washing water should not exceed a total volume of 350 c.c. and may run into a Jena beaker (low model) of 600 c.c. capacity.

The quantity of ammonia added corresponds in part to that necessary to precipitate the iron, and in part to the surplus recommended by Lunge. The washing of the ferric hydroxide precipitate is suitably carried out firstly twice on the filter with about 25 c.c. of warm water each time; thereafter the precipitate is transferred back to the beaker with about 50 c.c. water. After boiling with the water a couple of minutes, the precipitate is filtered upon the same filter, as before and finally washed with a further quantity of about 75 c.c. warm water.

7. Wash the ferric hydroxide precipitate into the precipitation beaker and dissolve by the addition of 5 c.c. hydrochloric acid, sp. gr. 1·12. Dilute the solution to about 100 c.c. and add, while warm, 5 c.c. barium chloride solution (110 grms./litre). If, after 24 hours, a weighable precipitate is deposited in the beaker, this should be separated by filtration and weighed separately or together with the barium sulphate precipitate in the filtrate from the iron precipitation.

If the washing of the iron precipitate is carried out as above described under 6, it is generally free from sulphuric acid.

8. Add to the filtrate from the ferric hydroxide precipitate a few drops of methyl-orange solution (0·1 grm./litre) and neutralise by adding hydrochloric acid sp. gr. 1·19 drop by drop. Add an excess of 1 c.c. of this hydrochloric acid and dilute to about 350 c.c.

About 2·5 c.c. hydrochloric acid are required for the neutralisation. This may suitably be added by means of a dropping-bottle as mentioned under 5.

9. Heat the solution to boiling, remove the burner and add at once while rapidly stirring 20 c.c. of a boiling barium chloride solution (110 grms./litre $BaCl_2$, $2H_2O$).

The barium chloride solution is added from a large test tube, bearing a mark for the exact measurement of 20 c.c. The solution in the beaker is firstly given a rapid rotary movement by quickly stirring three or four revolutions per sec, with a glass rod about 5 mm. thick, after which the barium chloride solution is added during continued stirring, as rapidly as possible, near the edge of the beaker and in the direction of the stirring.

10. Let the sample stand without heating until the precipitate has settled (at least ½ hour), and afterwards decant the clear solution through a close 9 cm. filter.—Test the filtrate with barium chloride to see that all sulphuric acid is removed.—Pour over the precipitate in the beaker 100 c.c. boiling water, stir rapidly, let the precipitate sink for about 5 minutes, and again decant the solution. Repeat this treatment twice again and then wash the precipitate on to the filter, where it is washed until the washings do not give any reaction for chlorine with silver nitrate.

If the filtrate from the barium sulphate precipitate is not completely clear, and if, after a few hours, a weighable deposit is found, a closer filter paper should be used, or else the sample should stand a longer time before filtering.

11. Transfer the filter paper with the precipitate, without previous drying, into a weighed platinum crucible (together with any filter and barium sulphate precipitate from the iron solution) laying the filter with its point upwards. Place the crucible upright on a triangle and carbonise the filter paper at so low a heat that the gases issuing from the crucible do not ignite Then lay the crucible inclined upon the triangle and burn off the carbon. Bring to red heat with the lid on until constant weight is obtained, using a Teclu or Meker burner (No. 2). A blast flame must not be used.

Barium sulphate × 0·1373 = sulphur.

12. The purity of the reagents is controlled by carrying out a special blank analysis, and the analysis results are corrected in regard to any sulphur found in the reagents.

METHOD B: SWEDISH-NORWEGIAN METHOD ACCORDING TO GYZANDER.

Method A is followed as far as point 4 inclusive and from point 9 onwards inclusive. Intermediate operations are carried out according to the following instructions.

5. Moisten the evaporation residue with 2 c.c. of hydrochloric acid (sp. gr. 1·19), and heat until all except the insoluble residue (gangue, silica, etc.) is dissolved. Wash the glass cover and dilute to about 100 c.c. with warm water. Filter off the insoluble residue and wash same with about 50 c.c. warm water. Let the filtrate run into a beaker of 600 c.c. capacity (low model).

In adding the hydrochloric acid a dropping-bottle may suitably be used, for which the number of drops of hydrochloric acid per c.c. has been determined.

6. Add to the beaker 20 c.c. solution containing 20 grms. hydroxylamine hydrochloride and 100 grms. ammonium chloride per litre and dilute to about 350 c.c.

The hydroxylamine hydrochloride reduces, upon subsequently bringing to boiling, any ferric salts to ferrous salts which are not occluded by the barium sulphate precipitate. The solution then becomes colourless.

The addition of ammonium chloride makes it possible to employ Lunge's method for the precipitation of the barium sulphate. In the absence of ammonium chloride a result about 0·2 per cent. too high is obtained upon precipitating according to Lunge's method, and upon precipitation by the addition, drop by drop, of barium chloride solution according to Gyzander's method, the results are 0·3 per cent. too low.

IV. THE CALCULATION OF THE SULPHUR ANALYSIS.

Upon each of the two ultimate samples, in addition to the determination of the sulphur content, a separate moisture analysis is made. For this 5 grms. are weighed into a weighing glass with a glass stopper (dimensions about 30 × 30 mm.) and dried for one hour at 105° C. The loss in weight is regarded as moisture.

After calculating the average values of sulphur and moisture analyses upon the ultimate samples the sulphur is then calculated upon the sample dried at 105° C. The result is given with one decimal.

The sulphur analysis must never be made upon samples which have been dried at a higher temperature than about 40° C.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Acid-soluble Phosphoric Acid in Eggs. L. Pine. (*J. Assoc. Off. Agric. Chemists*, 1924, 8, 57–70.)—For estimating the acid-soluble phosphoric acid in liquid whole egg and yolk, the following modification of the Chapin and Powick method (*J. Biol. Chem.*, 1915, 20, 97) is recommended. Fifty grms. of whole egg (or 25 grms. of yolk) are shaken vigorously at least every 10 minutes for 1 hour with 200 c.c. of hydrochloric acid solution containing 1 c.c. of the concentrated acid and 8 grms. of picric acid, in a 500 c.c. Erlenmeyer flask closed with a rubber stopper. The mixture is then filtered through a pleated filter paper, No. 24 (18·5) cm. in diameter, the filtration occupying not more than 45 minutes. From 125 to 150 c.c. of the filtrate are boiled with 10 c.c. of concentrated sulphuric acid and 10 c.c. of concentrated nitric acid in a 500 c.c. Kjeldahl flask, containing four glass beads, until white fumes appear; about 2 c.c. of concentrated nitric acid are then added, drop by drop, and the liquid again boiled until white fumes are evolved, this procedure being repeated four times and the final boiling continued for 10 minutes longer. The cold liquid is now boiled with about 25 c.c. of water until the brown fumes are expelled. While still hot, the solution is transferred to a 400 c.c. beaker, and the flask is washed with small quantities of hot water until the liquid in the beaker measures about 100 c.c. The phosphoric acid is then estimated by the official gravimetric method.

The total volume of solution in the extraction mixture is found by adding together 200 c.c., the volume of water contained in 50 grms. of whole egg or 25 grms. of yolk, and in 8 grms. of picric acid. Water is estimated in eggs in a vacuum at 55° C., and in picric acid in a vacuum over sulphuric acid. The percentage of water is, for whole eggs, between 75·4 and 63·19; for one-day old eggs, 73·57 on the average; for eggs kept for 11 months in cold store, 71·45 on the average; for yolk, between 47·16 and 54·55; for yolks from one-day old eggs, 47·56, and for yolks from eggs kept in cold storage for 11 months, 53·74, both on the average. The number of mgrms. of acid-soluble phosphorus pentoxide per 100 grms. of dry material is: (1) for one-day old eggs, from 76·9 to 95·5, the average being 83·4; (2) for market fresh eggs, from 72·0 to 93·6, the average being 83·3; (3) for eggs kept till the yolks stick to the shells, but are detachable by a rapid twist, the values are from 89·6 to 102·5, and 94·2; (4) for eggs kept in cold storage for 11 months, from 92·1 to 112·9, and 100·5; (5) for eggs kept until the yolks stick to the shells, but are detachable by several quick twists, from 88·2 to 114·8, and 101·2; for eggs with yolks non-detachable from the shells by twisting, from 97·8 to 127·5, and 108·0; for eggs showing "white rots," from 123·6 to 295·2, and 224·6, and for those showing "black rots," from 468·6 to 721·9, and 624·5. For the yolks of eggs of these classes, the values are: (1) From 93·9 to 111·9, and 100·5; (2) from 89·7 to 118·3, and 105·8; (3) from 97·2 to 122·6, and 110·6; (4) from 117·7 to 141·2, and 127·2; (5) from 106·6 to 120·4, and 114·1. T. H. P.

Rate of Disappearance of Sulphites in Raw Meat. F. H. Campbell. (*J. Soc. Chem. Ind.*, 1924, **43**, 297–298T.)—Sulphite was estimated in meat to which it had been added, and which was kept in hermetically sealed bottles, some at room temperature and others in a refrigerator, and the loss determined after varying periods of time. The greatest loss occurred during the first 7 days, but provided the temperature does not exceed 50° F. the loss is not sufficient to form a serious source of disagreement between analysts working on portions of the same sealed sample kept for varing periods up to 6 weeks, or even longer if the temperature is sufficiently low, but specific directions for the conditions of storing of such samples are needed. D. G. H.

The Bleaching of Pickled Herrings with Hydrogen Peroxide. O. Luning. (*Zeitsch. Unters, Nahr. Genussm.*, 1924, **48**, 120–122.)—The peculiar bright colour of the flesh in many samples of pickled herrings is due to the presence of hydrogen peroxide in the pickling fluid. One such fluid, sold under the name of "Blankalit," was found to contain 30 per cent. of hydrogen peroxide. Experiment has shown that it is often not possible subsequently to detect this preservative in the pickle after a time owing to the decomposition of the peroxide. Stale herrings, which are often used for pickling, have a peculiar oily flavour, probably due to atmospheric oxidation, and this is not removed by hydrogen peroxide. It is suggested that fat-soluble vitamin *A*, in which herrings are particularly rich, may be wholly or partly destroyed by the oxidising action of the peroxide.

H. E. C.

The Hydrocyanic Acid Fumigation of Ships and its effect on Food Material. P. Buttenberg and H. Weiss. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 104–119.)—The various methods of production of hydrogen cyanide for fumigating ships are described and discussed. The volume percentage used varies from 0·2 to 1·0, and the time of application is about 4 hours, followed by 12 to 18 hours' aeration. Great care is, of course, needed to avoid accidents. Experiments on cargoes of mixed foodstuffs, including cereals, fats, tea, coffee, chocolate, meat, tobacco and drugs, show that a minute quantity of hydrogen cyanide is actually retained by most substances, the amounts varying up to 2 mgrms. per 100 grms. for most commodities and up to 3 mgrms. in the case of raw coffee. It is concluded that there is no permanent deleterious effect on the foodstuffs, and that no perceptible odour or flavour is imparted thereto, except in the case of bulk tea, coffee and raw tobacco, and in the two latter the condition is readily restored by aeration. Only tea appears to be permanently, though slightly, injured, and on this point further investigation is needed.

A simple test for hydrocyanic acid vapour in air is made by suspending therein a filter paper impregnated with a mixed solution of benzidine and copper acetate. A blue colour is developed in the presence of prussic acid. H. E.C.

Composition of Soya Bean Oil. E. S. Wallis and G. H. Burrows. (*J. Amer. Chem. Soc.*, 1924, **46**, 1949–1953.)—The insoluble fatty acids of soya bean oil were analysed by the melting point method described by Twitchell (ANALYST, 1914, **39**, 448), and found to have the following composition:—Myristic acid, none; palmitic acid, 10 per cent.; stearic acid, 2 per cent.; arachidic acid, 1 per cent.; lignoceric, linolenic, linolic and oleic acids, 88 per cent. W. P. S.

Marine Animal Oils. Identity of Phocœnic Acid with Valeric Acid. E. André. (*Bull. Soc. Chim.*, 1924, **35**, 857–868.)—The acid prepared in 1817 by Chevreul from dolphin and porpoise oils and named phocœnic acid, is shown to consist, not of a mixture of butyric and caproic acids, as suggested by Lewkowitsch (*Chemical Technology and Analysis of Oils, Fats and Waxes*, 5th edition, 152) and by Fryer and Weston (*Technical Handbook of Oils, Fats and Waxes*, 1920, I., 37), but of isopropylacetic acid. T. H. P.

Chemistry of Grape Pigments. Anthocyanins in Clinton Grapes. R. J. Anderson and F. P. Nabenhauer. (*J. Biol. Chem.*, 1924, **61**, 97–107.)—The colouring matter occurring in Clinton grapes has been examined. Clinton is said to be the first cultivated variety of *Vitis riparia*, but it is believed that it also contains a derivative of *Vitis labrusca*. The pigment in Clinton grapes consists principally of a monoglucoside, anthocyanin, and is identical with the anthocyanins isolated from Norton and Concord grapes. The anthocyanin chloride, $C_{23}H_{25}O_{12}Cl$, did not separate in definitely crystalline form, but the picrate, $C_{23}H_{24}O_{12}.C_6H_2(NO_2)_3OH$, crystallised in beautiful bright red prisms or needles. The glucoside is easily hydrolysed by boiling with 20 per cent. hydrochloric acid, yielding 1 molecule each of glucose and anthocyanidin chloride, $C_{17}H_{15}O_7Cl$, the latter crystallising from the hot solution in well-formed prisms. The anthocyanidin chloride has the same percentage composition as oenidin chloride, but it differs from this substance in that it contains a lower percentage of methoxyl. It consists largely of a monomethyl ether of delphinidin, but the values for methoxyl were too high, indicating that it contained some of the dimethyl ether of that substance. It differs in its colour reaction with ferric chloride from the anthocyanidin from Norton and Concord grapes, probably owing to a difference in position of the methoxyl groups in the gallic acid part of the molecule. Results indicate that the methyl groups are attached to the gallic acid part of the molecule and not to the phloroglucinol. The absorption spectra of anthocyanin chloride and anthocyanidin chloride consist of one broad band extending from the yellow into the blue (*cf.* ANALYST, 1924, 39).

P. H. P.

Lead Number of Vanilla Extracts. C. A. Clemens. (*J. Assoc. Off. Agric. Chemists*, 1924, **8**, 79–82.)—Comparison is made of the estimation of lead as sulphate and as chromate in the determination of the lead numbers of 36 samples of vanilla extract by the official Winton method and by the Wichmann modification (ANALYST, 1921, **46**, 333). With the former method, the values obtained ranged from 0·28 to 0·71 or from 0·27 to 0·73, according to whether the sulphate or chromate method was used; after elimination of the only two values below 0·40, which resulted from incomplete extraction of the beans, the mean values became 0·53 and 0·55 respectively. The Wichmann lead numbers ranged from 0·46 to 0·98, or from 0·47 to 1·06 for the sulphate and chromate methods, respectively, the average values being 0·78 and 0·83; the excellent coagulation of the colloidal lead precipitate effected by this method is due to the combined effects of the temperature, dilution and alcohol employed. When the lead is estimated as chromate, the procedure is as follows: Ten c.c. of the filtrate (*loc. cit.*) are heated in a 400 c.c. beaker with 2 c.c. of glacial acetic acid, 25 c.c. of water, and 25 c.c. of approximately 0·1 *N* potassium dichromate solution until the precipitate becomes orange-red; the heating should commence immediately the dichromate has been added, and a stirring rod should not be put into the liquid while this is boiling. The precipitate is transferred to a weighed Gooch crucible by means of hot water, and washed thoroughly, first with hot water and then with a little alcohol and a little ether; after being dried in a vacuum oven at 100° C. for 30 minutes, the lead chromate is weighed; multiplication of the weight by 12·82 gives the lead number. If this procedure is coupled with the Wichmann method, the lead number may be determined in 1½ hours, at most.

T. H. P.

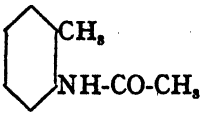
Detection of Extract of Atractylis gummifera in Liquorice Extract. U. Giuffrè. (*Giorn. Chim. Ind. Appl.*, 1924, **6**, 375–377.)—If 100 grms. of the suspected liquorice extract are treated by the author's method (ANALYST, 1923, 48, 30), the presence of 1 per cent. of the extract of *Atractylis gummifera* is sufficient to show clearly the colour reaction of the atractyligenin. Bertolo's method (*Giorn. Chim. Ind. Appl.*, 1923, 5, 391), based on extraction of the potassium atractylate by means of absolute alcohol and subsequent precipitation of the sulphonic sulphur as barium sulphate, involves more manipulation than the above method and is less sensitive and less certain. T. H. P.

Certain Reactions of Carrageen (*Chondrus crispus*). E. Justin-Mueller. (*J. Pharm. Chim.*, 1924, 30, 154–161.)—Ten grms. of the seaweed were boiled for 2 hours with 1000 c.c. of water, and the solution brought to its original volume, strained through linen and left to cool. The clear supernatant liquid was gummy, limpid and neutral, and gelatinised on removal of 15 to 20 c.c. of water per litre. This liquid was used cold with the various reagents and colouring matters, a sample of untreated liquid being taken for comparison. With methyl violet, magenta, malachite green, picric acid, no change occurs, whilst a marked flocculation takes place with methylene blue and safranine, even although these, together with all the colouring matters tried (except picric acid) were of basic nature. The flocculating action of a decoction of this seaweed thus serves as a differentiating reagent for organic compounds. D. G. H.

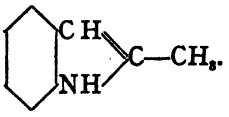
Biochemical, Bacteriological, etc.

Catalytic Action of Copper in the Destruction of Anti-scorbutic Vitamin. A. F. Hess and M. Weinstock (*J. Amer. Med. Assoc.*, 1924, 82, 952–956; *Chem. Abstr.*, 1924, 18, 1690–1691.)—Experiments with guinea pigs showed that the anti-scorbutic vitamin is readily destroyed by oxidation, especially when the oxidation process takes place in association with heat. This sensitiveness leads to a partial loss of this factor in the pasteurisation of milk. The gradual decrease of vitamin in the ageing of foods, such as vegetables, is to be interpreted in this way. Catalysis greatly increases the velocity of oxidation and furthers the destruction of this vitamin. It was found that when as little as 2·5 mgrms. of copper per litre were added to milk, the anti-scorbutic vitamin was appreciably diminished in the course of heating. Although copper does not dissolve to this extent in properly supervised pasteurisation, such contamination readily occurs when proper care is not taken of the plant. The risk is still greater in the process of condensing milk. A similar danger exists with regard to the contamination of butter and cream, as vitamin A, is highly sensitive to the destructive action of oxidation. At present, contaminations of foods with copper or other metals are judged by the standards of toxicology. This criterion is inadequate, for even non-toxic traces of these substances are sufficient to bring about the catalytic destruction of vitamins.

Sodamide as a Dehydrating Agent. Preparation of Indol, Methyl-Indol and its Homologues. M. A. Verley. (*Bull. Soc. Chim.*, 1924, 35, 1039–1040.)—To remove one molecule of water from acetyl-ortho-toluidine, (I) and obtain methyl-indol (II) appears simple but all the usual dehydrating agents give entirely negative results. Sodimide, however, in sufficient excess, gives the desired



(I).



(II).

result. One part of acetyl-*o*-toluidine and 2½ parts of roughly ground sodamide are placed in a metal apparatus and gently warmed, when the mixture begins to melt with a rather violent evolution of ammonia. The temperature of the semi-liquid mass is then raised to 250° C. and heating is continued until the evolution ceases. After being left to cool the product is taken up with water and steam-distilled. An oil collects which forms two portions, one of regenerated *o*-toluidine and the other of methyl-indol, which crystallises immediately. After recrystallisation from petroleum spirit it melts at 59° C. The *o*-toluidine is formed by saponification. The higher homologues of methyl-indol are formed similarly. Indol can be obtained in this way from formyl-*o*-toluidine, but certain precautions are necessary, since the reaction takes place with explosive violence.

P. H. P.

Preparation of Yeast Nucleic Acid as a Magnesium Compound. E. J. Baumann. (*J. Biol. Chem.*, 1924, **61**, 1–4.)—Other methods for the preparation of large quantities of yeast nucleic acid are either laborious or yield products contaminated with protein. Steudel and Peiser (*Z. physiol. Chem.*, 1922, **120**, 292) showed that 3 per cent. sodium hydroxide splits off guanylic acid from yeast nucleic acid at room temperature. The author gives details of a simple, inexpensive method of preparation for which only a very mild and brief treatment of the yeast with weak alkali is used. The crude product contains no protein. The process depends upon: (1) The concept that nucleic acid exists in yeast as a nucleoprotein, and that nucleoprotein is a type of acid protein which is easily separated into its components, nucleic acid and protein, by alkali, forming the alkali salt of the nucleic acid and meta-protein; (2) the solubility of sodium nucleate in dilute acetic acid; (3) the precipitation of nucleic acid as a magnesium salt by magnesium sulphate and hydrochloric acid. The ratio of phosphorus to magnesium indicates that the acid is precipitated as a mixture of the mono- and di-magnesium salts. Attempts at purification have not yielded very satisfactory analytical results. A table is given of analyses of different air-dried unpurified preparations.

P. H. P.

Estimation of Phenols in Blood. R. C. Theis and S. R. Benedict. (*J. Biol. Chem.*, 1924, **61**, 67–71.)—A method is described by which phenols can be estimated directly on the Folin-Wu filtrate. Diazotised *p*-nitraniline base as described by Moir in his phenol test (*J. South African Inst.*, 1922, **5**, 8) is the reagent used as it is more specific for phenols than is the phosphotungstic-molybdic reagent. It is prepared as follows: *p*-Nitraniline base (1·5 grms.) is dissolved in

500 c.c. of water with 40 c.c. of concentrated hydrochloric acid. Twenty-five c.c. of this solution are diazotised by 0·75 c.c. of 10 per cent. sodium nitrite solution. The diazotised solution only keeps for 1 day. This reagent is sufficiently delicate for phenol estimations in blood filtrates, yielding an orange to red coloration with phenols. It is necessary to dilute the blood only 1 to 5, instead of 1 to 10, as in the regular Folin-Wu procedure. The addition of a colloid (gum arabic) prevents turbidity of the reagent with blood filtrates. The reaction takes place in solutions of very weak acidity. Uric acid reacts so feebly with the nitraniline reagent that its removal is unnecessary. Details of the method and tables of results are given. Figures for free phenols in the blood were obtained ranging from 1 to 2 mgrms. per 100 c.c. Conjugated phenols probably occur in small quantity in some bloods, but are not demonstrable in all bloods. Pelkan's criticism of this is discussed. The figures obtained by this method on 20 bloods average 0·4 mgrm. per 100 c.c. lower than the figures of the Rakestraw method on the same bloods. The application of the nitraniline method to urine is being studied. P. H. P.

Method for Cholesterol Estimation in Blood. S. L. Leiboff. (*J. Biol. Chem.*, 1924, **61**, 177–180.)—A new rapid and accurate method is described for estimating cholesterol in blood. A filter paper disc is dropped into an extraction tube so that it lies above the constricted portion of the tube, which contains about 5 c.c. of chloroform. Diagrams of the disc and tube are given. By means of a pipette 0·25 c.c. of oxalated blood are placed on the disc and absorbed, then the tube is attached to a reflux condenser and immersed in hot water and extracted for 30 minutes. The tube is next detached from the condenser and, after the removal of the disc, is cooled by immersion in cold water. Chloroform is added exactly to the 5 c.c. mark. In a similar extraction tube are placed 5 c.c. of a standard solution containing 0·4 mgrm. of cholesterol per 5 c.c., made by dissolving 40 mgrms. of pure dry cholesterol in 500 c.c. of pure dry chloroform. To each tube are added 2 c.c. of pure anhydrous acetic anhydride and 0·1 c.c. of concentrated sulphuric acid. The tubes are inverted twice to mix the contents and then, stoppered, are placed in cold water for half a minute. After being left in a dark place for 10 minutes the readings are taken in a colorimeter. $S/R \times 160$ $=$mgrm. per 100 c.c. of blood where $S=$reading of standard, and $R=$reading of unknown. Experiments described and tables given show that the extraction is complete in 30 minutes. Results showed slightly higher figures than by the method of Myers and Wardell (*J. Biol. Chem.*, 1918, **36**, 147), where losses are incurred by the transfer of material. P. H. P.

Modification of Molybdic Method for the Estimation of Inorganic Phosphorus in Serum. S. R. Benedict and R. C. Theis. (*J. Biol. Chem.*, 1924, **61**, 63–66.)—A modification of the molybdic method for the estimation of inorganic phosphorus in serum has been devised in an attempt to avoid turbid solutions, and to obtain a more intense and stable colour. The colour produced is about three times as intense as that obtained by the method of Briggs (*J. Biol. Chem.*, 1922, **53**, 13), and the colour of a standard solution has remained unchanged for several days. If the blood available is limited, less than stated may be used. In 12 cases where phosphates were added to serum an average of 103 per cent. was recovered. A table shows that the results obtained by this method compare favourably with those by Briggs' method. The method is not applicable to whole blood or to the serum or plasma from badly haemolysed blood, since the heating and strong acid will cause hydrolysis of the organic phosphorus compounds contained in the corpuscles. Estimations in blood serum are carried out as follows:—To 2 c.c. of serum, diluted with a little water in a volumetric flask, 4 c.c. of 20 per cent. trichloracetic acid are added, and the whole is diluted to 10 c.c., left for 10 minutes and filtered through ashless filter paper. Five c.c. of the filtrate are placed in a tube and 3 c.c. of water added, then 1 c.c. of diluted molybdic acid reagent (to 20 grms. of pure molybdic acid (MoO_3) in a flask add 25 c.c. of 20 per cent. sodium hydroxide solution and warm gently to dissolve the acid, cool and dilute to 250 c.c., filter if necessary and dilute a small quantity of this reagent 1:1 with concentrated sulphuric acid as needed), and 1 c.c. of a solution containing 15 grms. of sodium bisulphite and 0·5 grm. of hydroquinone in 100 c.c. This is mixed, loosely stoppered, and placed in a boiling water bath with a simultaneously prepared standard solution of potassium biphosphate containing 0·025 mgrm. of phosphorus in 5 c.c. which has been treated similarly to the blood filtrate. The standard is prepared from a stock solution (containing 0·4394 grm. of dry potassium biphosphate in 1 litre of water and preserved with chloroform) by diluting 5 c.c. of stock solution to 100 c.c. with water and preserving with chloroform. By this standard a satisfactory degree of accuracy for phosphorus contents between 0·05 and 0·0125 mgrm. is shown. It is noted that no acid-soluble organic phosphorus is found in serum after hydrolysis with concentrated sulphuric acid. P. H. P.

Toxicological, Forensic, etc.

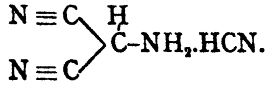
Case of Poisoning by Fluosilicic Acid. Lührig. (*Chem. Zeit.*, 1924, **48**, 613–614.)—Attention is drawn to the highly toxic nature of fluosilicic acid, and a fresh case of fatal poisoning is described, in which, through an error in dispensing, a beetle powder containing this acid was given to a boy in mistake for powdered senna. *Post-mortem* examination showed the presence of the acid in the blood, stomach, intestines, kidneys, and liver; the acid was identified by igniting the tissues, after drying, with lime water, and then observing the etching of glass in the usual way, after adding sulphuric acid to the ash. H. E. C.

Case of Acetanilide Poisoning. Estève. (*Bull. Soc. Pharm. Bordeaux*, 1923, No. 4; *Ann. Chim. anal.*, 1924, **6**, 272–274.)—After taking 0·5 grm. of acetanilide a day for about a month the patient became comatose, with pale face, cold extremities and cyanosed lips. Acetanilide passes into the urine as para-aminophenol sulphate and in this case was present 8 days after the patient had stopped taking the drug. It was identified by evaporating to dryness the ethereal extract of the urine acidified with phosphoric acid, treating the residue with hydrochloric acid, boiling, and, after cooling, adding 1 c.c. of water saturated with phenol, and a few drops of a saturated solution of calcium hypochlorite, shaking after the addition of each drop. A red colour was produced, changing to blue with ammonia. The accumulation of the drug was probably due, in part, to some failure of the excretory organs. It was conclusively shown that cyanosis was not due to the presence of methaemoglobin and haematin in the blood, or the general poisoning to the presence of aniline as an impurity in the acetanilide.

D. G. H.

Fate of Morphine in the Organism. R. Fabre. (*J. Pharm. Chim.*, 1924, **30**, 183–187.)—Morphine was found to be unaltered by blood or during the course of pancreatic and peptic digestion, but appeared to be changed in the liver. Twenty c.c. of a 1 per thousand morphine hydrochloride solution was injected into a rabbit's liver under physiological conditions, left for 5 minutes; replaced by a further 20 c.c., which was again left for 5 minutes; the operation repeated with the first 20 c.c., and so on. The proportion of morphine in the solution steadily diminished until at the 6th circulation it was found to be absent from the solution and also from the liver itself. D. G. H.

Toxicity of a Polymer of Hydrocyanic Acid. C. Bedel. (*J. Pharm. Chim.*, 1924, **30**, 189–193.)—A crystalline substance was isolated from azulmic acid representing 2 per cent. of its total weight, and agreeing with the formula



When this substance was administered to female guinea pigs by mouth the fatal dose was found to be as much as 0·75 grm. per kilo. of animal. *Post-mortem* analysis showed that free hydrocyanic acid was absent from the intestines, liver and heart, and only a very small quantity was detected in the stomach, whilst the kidneys gave a positive reaction. The symptoms of poisoning were similar to those recorded for hydrocyanic acid, but more delayed, and it is probable that slow depolymerisation occurs. Sodium hyposulphite appears to exercise no antidotal action on the polymer of cyanic acid. D. G. H.

Water Analysis.

Estimation of Nitrate in Water by the Frerichs' Method. A. Massink. (*Chem. Weekblad*, 1924, **21**, 421.)—Frerichs' method of estimating nitrates in water (*Arch. Pharm.*, 1903, **241**, 47) has been found to be inaccurate, the results being too high when alkali carbonates are present. During the neutralisation of the water with sulphuric acid, alkali sulphates are formed and these, on evaporation of the solution to dryness with hydrochloric acid, form bisulphates according to the equation $Na_2SO_4 + HCl \rightarrow NaHSO_4 + NaCl$. Thus there would be an increase in the chlorine ions and also in the acidity, both factors contributing to an increase of the titre of the standard silver nitrate. W. S. S.

Agricultural Analysis.

Toxicity of Cotton-Seed and its Gossypol Content. E. W. Schwartze and C. L. Alsberg. (*J. Agric. Res.*, 1924, **28**, 173–189.)—A comparison of the toxicity of pure gossypol with that of Trice, Lone Star, Durango, and Egyptian cotton-seeds has been made. The samples contained 0·41, 0·52, 0·98, and 1·12 per cent. of gossypol respectively, and it is found that injection of the gossypol in arachis oil solution showed a toxicity corresponding with the gossypol content of the different varieties of kernels which shows that the poisonous quality of cotton-seed is due to the gossypol content. The variation in the amount of gossypol in cotton-seed from different producing regions explains the variation in the incidence of cotton-seed poisoning in cattle. Details are given of various adequate diets given to the rats and the physiological effects of the gossypol. The analyses of the cotton-seed kernels show the gossypol to be approximately proportional to the ether extract and inversely proportional to the nitrogen content. For the method of isolation see ANALYST, 1924, 47.

H. E. C.

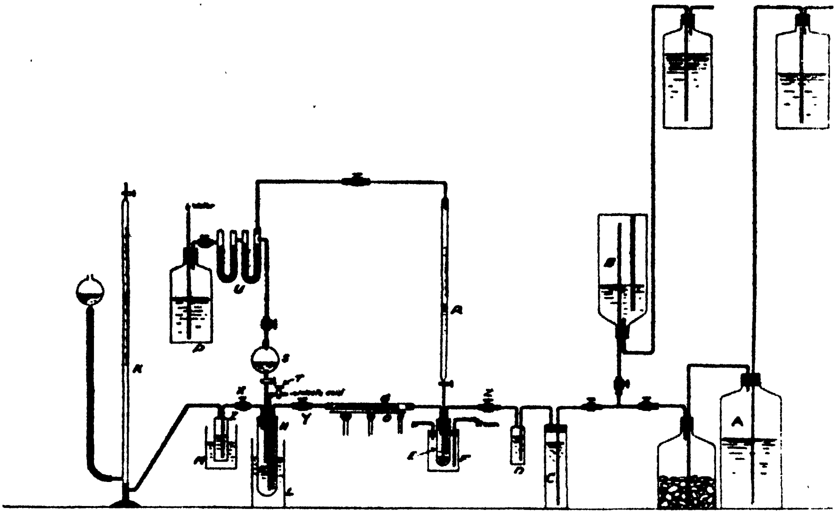
Pharmacology of Gossypol. E. W. Schwartze and C. L. Alsberg. (*J. Agric. Res.*, 1924, **28**, 191–197.)—Administration of gossypol over long periods to cats and rabbits in small doses produces chronic poisoning, the principal symptoms of which are paralysis, with nerve degeneration, shortness of breath, cardiac hypertrophy, oedema of the lungs and effusion into the serous cavities. Pure gossypol is capable of producing all the manifestations characteristic of the cotton-seed poisoning of farm animals, so that the conclusion is warranted that gossypol is the principal causative agent. The only other possible toxic substance in cotton seed kernels could be some decomposition product of gossypol. H. E. C.

Significance of Urea in Shark Meal. D. B. Dill. (*J. Assoc. Off. Agric. Chemists*, 1924, **8**, 70–75.)—A sample of shark meal sold with a guarantee of 70 per cent. of protein was found to contain 11·28 per cent. of total nitrogen and 1·93 per cent. of nitrogen in the form of urea. The discrimination between protein nitrogen and urea nitrogen is probably of no significance if the meal is to be used as fertiliser, but, as a food, urea nitrogen is of less value than protein nitrogen for ruminants, and probably of no value for other animals. T. H. P.

Organic Analysis.

Bromimetric Estimation of Thiocyanate. A. Kurtenacker and H. Kubina. (*Zeitsch. anal. Chem.*, 1924, **64**, 442–444.)—Titration with bromine water (König, *J. prakt. Chem.*, 1911, **84**, 558), as well as with bromate—with and without addition of bromide—gave variable results. The estimation of thiocyanate by these methods is unreliable. W. R. S.

Estimation of Water in Mixtures of Ether, Alcohol and Water. R. K. Newman. (*J. Soc. Chem. Ind.*, 1924, 43, 285T.)—The principle of the method described depends on the fact that the acetylene produced by the action of calcium carbide on such mixtures is not absorbed by 98 per cent. sulphuric acid to any appreciable extent, whereas both ether and alcohol are readily absorbed. The estimation is carried out in the apparatus shown in the diagram all air being previously replaced by carbon dioxide. The mixture to be examined is run from the burette R into the boiling-tube E, the prolonged end of the burette dipping under mercury at the bottom of E and thus preventing the escape of ether vapour during the estimation. Tube E is heated gradually by introducing steam into the jacket F, and the vapours are led over the calcium carbide in the combustion-tube G, which can be warmed if necessary. The mixed gases are then made to bubble through two gas-washing bottles containing 98 per cent. sulphuric acid, which absorb all ether and alcohol. The unabsorbed acetylene and carbonic acid are then passed into the nitrometer K, which contains strong aqueous potassium hydroxide solution, and the volume of acetylene can then be read off. A correction has to be applied, since carbonic acid, either from cylinders or from marble and acid, gives a small amount of a gas not absorbed by potassium hydroxide. The apparatus is designed so as to allow of a steady pressure of carbonic acid being maintained, and also so that for successive estimations the spent sulphuric acid can be replaced without admitting air into the apparatus. In testing made-up mixtures of known composition it was found that ether dried over sodium for 6 months still contained small amounts of water (about 0·0120 grm. of water per 10 c.c. of ether). After allowing for this and plotting a curve for c.c. of acetylene evolved with definite amounts of added water, it is seen that when the water content is very low the test appears to lose sensitiveness, owing probably to the difficulty of bringing all the water vapour into contact with the calcium carbide.



R. F. I.

Fixed Oil of Orris. P. Langlais and J. Goby. (*Perfumery and Essential Oil Rec.*, 1924, **15**, 311.)—During the preparation of some absolute oil of Florentine orris, the authors obtained a portion entirely soluble in sodium carbonate solution and composed of organic acids with a boiling-point below that of myristic acid and a melting point below 28° C. Distillation of these acids at 40 mm. pressure showed them to consist of a very complex mixture, the first fraction passing over at 130° C. and the last at 215° C., with no distinct steps between these points. The last of four fractional distillations was again distilled in six fractions at 40 mm. pressure. No. 1 (150°–151° C.) consisted of a small quantity of liquid; No. 2 (162°–163° C.), small quantity of liquid; No. 3 (174°–175° C.), important quantity of solid; No. 4 (185°–186° C.), small quantity of liquid; No. 5 (198°–200° C.), important quantity of solid; and No. 6 (210°–212° C.), small quantity of solid. This method of separation revealed the presence of the six saturated aliphatic acids: caprylic, pelargonic, capric, undecylic, lauric, and tridecylic, and also of benzoic acid. These were all identified by such methods as the preparation of the silver salts, with subsequent determination of the combining weight and m.pt. of the purified fatty acids and also the m.pt. of their anilides. The authors point out that the acids with 9, 11, and 13 carbon atoms, respectively, have not before been found in nature, and have up to now only been prepared synthetically. R. F. I.

Characteristics of Japanese Tung Oil. I. Miura. (*J. Chem. Ind.* (*Japan*), 1923, **26**, 316–321; *Chem. Abstr.*, 1924, **18**, 1398.)—There are four species of tung oil trees in Japan, *viz.* (*a*) Japanese tung oil tree, *Aleurites cordata*, (*b*) Chinese tung oil tree, *A. Fordii*, (*c*) Kangtong tung oil tree, *A. montana*, and (*d*) Bakoly (or tropical) tung oil tree, *A. moluccana*. Of these, (*a*) is grown all over the interior of the country, and the others in Formosa. The following results were obtained:

	a.	*b.*	*c.*	*d.*
Oil contents, in seeds, per cent. ..	37·8	44·1	37·4	23·6
,, in endosperm, per cent.	59·1	66·3	59·8	70·7
Time for drying at 95° C., minutes	65	55	60	270
Specific gravity at 15° C. ..	0·9340	0·9488	0·9372	0·9267
Acid value	0·47	1·52	0·59	0·80
Saponification value	194·8	196·7	194·3	195·1
Iodine value (Wijs)	151·4	157·2	154·8	146·3
Refractive index, n^{25}_{D}	1·5065	1·5210	1·5147	1·4785
Reichert-Meissl value	0·39	1·10	0·35	0·71
Unsaponifiable matter, per cent.	0·41	0·59	0·99	0·97

Lignoceric Acid. P. A. Levene, F. A. Taylor and H. L. Haller. (*J. Biol. Chem.*, 1924, **61**, 157–161.)—Lignoceric acid is a constituent of cerebrosides and of sphingomyelin. Samples of it freshly prepared from two different sources, *viz.* from arachis oil and from cerebrosides isolated from the brains of cattle, had exactly the same melting point of 80·5–81° C. and a mixture of the two substances also melted at the same temperature. Thus there is no reason to revise the older conclusions that lignoceric acid is not identical with the normal tetracosanic acid, although Brigl and Fuchs (*Z. physiol. Chem.*, 1922, **119**, 280) stated it to be a mixture of normal tetracosanic acid melting at 85° C. and a second tetracosanic acid melting at 75° C. Possibly beechwood tar is not a satisfactory material for the preparation of unaltered lignoceric acid. The acid from arachis oil was crystallised three times from pyridine, melted over dilute hydrochloric acid on a water bath and crystallised from acetone. It then melted at 78·5–79·5° C. Four crystallisations of this product from ether gave the pure acid melting at 80–81° C. The pure acid was then fractionated and re-crystallised several times from ether, benzene, ethyl alcohol, toluene and pyridine without any change in the melting or solidification point. The acid from the cerebroside, "cerasin," after many recrystallisations from pyridine and other solvents, gave a m.pt. of 81° C, and then did not change. Finally samples of each acid were converted into the ethyl esters and saponified back again. After recrystallisations the melting points were again 81° C. and did not change. P. H. P.

Separation of Unsaturated from Saturated Sterols. R. J. Anderson and F. P. Nabenhauer. (*J. Amer. Chem., Soc.*, 1924, **46**, 1957–1960.)—The mixed sterols contained in the endosperm of wheat and maize may be separated from each other by dissolving the mixture in carbon tetrachloride and treating the solution with acetic anhydride and sulphuric acid; the unsaturated sitosterol combines with the sulphuric acid forming a stable soluble compound having a bluish or purple-green colour. The organic solvent is separated from the acid layer, washed with a small quantity of water, and evaporated. The residue is saponified and crystallised from alcohol in order to obtain the saturated dihydrositosterol. W. P. S.

Quantitative Estimation of Lignin in Wood Pulp. H. Wenzl. (*Papier fabr.*, 1924, **22**, 101–106; *Chem. Abstr.*, 1924, **18**, 1749.)—A new method is described based on the use of a mixture of phosphoric anhydride and hydrochloric acid as reagent. Thirty grms. of phosphoric anhydride are added to 100 c.c. of concentrated hydrochloric acid (sp. gr. 1·19) contained in a beaker immersed in cold water. Twenty-five c.c. of this reagent are added to 1 grm. of the air-dry pulp (without previous extraction), and the mixture stirred, put into a stoppered bottle, which is then kept for 30 minutes in a water bath at 30° C. As a rule, after this time, the cellulose is completely hydrolysed. The reaction mixture is diluted with hydrochloric acid (sp. gr. 1·19), which has previously been heated at 30° C. for an hour, and filtered on to a Gooch crucible containing an asbestos mat, which has been moistened with acid. The residue is washed, first with hydrochloric acid, and then with hot water until free from acid, and dried at 110°–115° C. It may then be calcined in the same crucible. The results (obtained in 4 to 5 hours) agree closely with those given by the Krull modification of the hydrochloric acid method.

Inorganic Analysis.

2-4-Dinitrosoresorcinol as a Sensitive Reagent for Ferrous Salts. M. Goldstück. (*Chem. Zeit.*, 1924, **108**, 629.)—A solution of 2-4-dinitrosoresorcinol in boiling water is prepared, of a concentration sufficient to give a brown colour. A few drops of this are added to the solution to be tested, which may be slightly acid or slightly alkaline. The formation of a greenish-blue precipitate indicates the presence of ferrous salts. If only traces of these are present a green colour is produced. The presence of free mineral acid or strong alkali interferes with the test, although the precipitate once formed is insoluble in these. Tartaric and acetic acids may be present, but not oxidising agents such as nitric acid or hydrogen peroxide. Copper salts must be absent. The delicacy of the reaction is such that the presence of 0·00002 grm. of ferrous iron in 10 c.c. can be detected, thus showing this reagent to be more delicate than potassium ferricyanide.

R. F. I.

Ferric Oxalate. Application to the Separation of Iron and Calcium. J. Barlot. (*Bull. Soc. Chim.*, 1924, **35**, 1026–1030.)—The difficulty of obtaining precipitated ferric hydroxide free from entangled calcium carbonate may be avoided if the calcium alone is first precipitated, the iron and aluminium being left in solution. Aluminium oxalate is soluble in acetic acid, and the double ferric calcium oxalate is decomposed by acetic acid in presence of ammonium oxalate, with formation of a soluble double oxalate of iron and ammonium, the precipitate then containing only the whole of the calcium as oxalate.

The hydrochloric acid containing the aluminium, iron, calcium, and magnesium, is boiled with a little nitric acid to oxidise the iron, neutralised as exactly as possible with ammonia, and treated with a dew drops of ammonium chloride solution and with ammonium oxalate. Acetic acid is then added in such quantity that the yellowish-brown precipitate becomes perfectly white and is composed solely of calcium oxalate; the calcium is estimated in the usual way. Addition of ammonia solution to the liquid at 80–90° C. now precipitates the iron and aluminium hydroxides in a pulverulent, non-gelatinous form. The magnesium is precipitated in the usual way. T. H. P.

Gravimetric Estimation of Copper. E. Wilke-Dörfurt and U. Rhein. (*Zeitsch. anal. Chem.*, 1924, **64**, 380–388.)—Precipitated cuprous sulphide may be weighed as such (Rose's method) or roasted to oxide. The latter procedure is more convenient for technical purposes, but it generally gives a slightly high result. This is not necessarily due to an admixture of sulphate, but to carbon derived from the charred filter paper, which becomes firmly coated with copper oxide. The positive error disappeared when the cuprous sulphide was collected on a porous porcelain crucible (ANALYST, 1924, 454) and ignited to oxide. The use of the porous crucible also simplifies and shortens the estimation. W. R. S.

Separation of Copper from the Platinum Metals. W. H. Swanger and E. Wichers. (*J. Amer. Chem. Soc.*, 1924, **46**, 1814–1818.)—Copper may be separated quantitatively from platinum, palladium, iridium, and rhodium by means of ammonium thiocyanate if the solution contains sulphur dioxide in quantity sufficient to prevent the platinum metals from forming thiocyanates, and if the solution is sufficiently acid to prevent precipitation of the rhodium double sulphite. The procedure recommended is as follows:—The solution, which should measure about 200 c.c., may contain several grms. of the platinum metals, but not more than 0·1 grm. of copper; it is treated with successive 20 c.c. portions of saturated sulphur dioxide solution until, after being digested for thirty minutes on a water-bath, the mixture still smells strongly of sulphur dioxide and has a yellow colour. After cooling, the mixture is nearly neutralised with sodium hydroxide solution, and 10 drops of concentrated hydrochloric acid are added. Ammonium thiocyanate solution (2 grms. of the salt dissolved in saturated sulphur dioxide solution) is then added, drop by drop, 10 cc. being required for each 0·05 grm. of copper present. After eighteen hours the precipitate is collected on a filter, washed with dilute sodium chloride solution, and the filter and precipitate are digested on a water-bath with dilute nitric acid, the filter-paper pulp is separated by filtration, and the copper in the filtrate is re-precipitated as described above. The paper pulp retains the platinum metals which contaminated the first copper precipitate; these metals may be recovered by digesting the pulp with *aqua regia* and adding the solution to the main filtrate. The filtrate from the second copper precipitate also contains a small amount of platinum metals. The final precipitate of cuprous thiocyanate is collected on a filter, washed with very dilute ammonium chloride solution, ignited, reduced in an atmosphere of hydrogen, and the resulting metallic copper is weighed. W. P. S.

Separation of Rhodium from Platinum. E. Wichers. (*J. Amer. Chem. Soc.*, 1924, **46**, 1818–1833.)—The following method is recommended for the separation of rhodium from platinum in hydrochloric acid solution from which other heavy metals have been removed and which does not contain ammonium salts, alkali sulphates or acetates. The solution is treated with sodium hydroxide solution until yellow rhodium hydroxide begins to precipitate, and the volume of the solution is then adjusted so that more than 1 grm. of the metals is present per 100 c.c. Ten c.c. of a mixture of equal volumes of 9·0 per cent. barium chloride solution and 3·6 per cent. sodium carbonate solution are then added, the solution is boiled for two minutes and filtered, and the precipitate washed with hot 2 per cent. sodium chloride solution and re-dissolved in dilute hydrochloric acid. If carbon dioxide is not evolved on the addition of the acid, too little barium carbonate suspension has been used, and the platinum solution should be given a further treatment. The hydrochloric acid solution is filtered, diluted to 200 c.c., treated with sodium hydroxide and barium carbonate as before, boiled for three minutes, and filtered. If more than 1 per cent. of rhodium is present, a third separation may be necessary. The final hydrochloric acid solution, containing 25 c.c. of dilute hydrochloric acid (1:4) is diluted to 150 c.c., heated to 100° C., and treated for thirty minutes at this temperature with hydrogen sulphide. The precipitated rhodium sulphide is collected on a filter, washed with dilute ammonium chloride solution, ignited in a porcelain crucible, reduced in a current of hydrogen, and the reduced metal cooled in an atmosphere of hydrogen and weighed. W. P. S.

The Hydrostrychnine Reagent for the Estimation of Nitrite and Nitrate. I. M. Kolthoff. (*Chem. Weekblad,* 1924, **21,** 423.)—The hydrostrychnine reagent is prepared as follows: Five c.c. of a 1 per cent. aqueous solution of strychnine is mixed with 5 c.c. of hydrochloric acid (sp. gr. 1·19), and from 4 to 5 grms. of granulated zinc are added. The mixture is placed in a boiling water-bath for 5 to 10 minutes, cooled, and, after one hour's standing, the clear supernatant liquid is decanted. To prevent the reagent becoming coloured through oxidation by the air, it is advisable to leave it in a loosely stoppered bottle in the presence of a small quantity of zinc amalgam. The best method of using the reagent is that given by Denigès (*cf.* ANALYST, 1911, **36,** 432).

The reagent may be used for the quantitative estimation of nitrate, but it must be borne in mind that the reaction is not specific for nitrates, as it is also given by nitrites and oxidising agents. Traces of iron also give a coloration, and it is necessary to remove the iron as hydroxide. When nitrite is present in the water being examined for nitrate, the tint obtained must be compared with that given by a standard nitrate solution containing the same quantity of nitrite as the water under examination. W. S. S.

Physical Methods, Apparatus, etc.

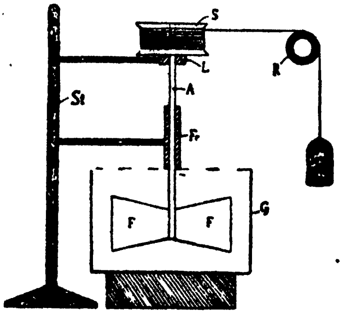
Use of Paper Pulp in Analytical Filtrations. E. Wilke-Dörfurt and E. Locher. (*Zeitsch. anal. Chem.,* 1924, **64,** 436–441.)—Dittrich's method of filtering ferric and aluminium hydroxides by mixing them with filter pulp occasionally leads to low results, the cause of which was investigated. Iron (aluminium) complexes, not precipitated by ammonia, are formed when a solution containing filter pulp and sulphuric as well as nitric acid is evaporated. If the evaporation is prolonged on the water-bath until the nitric acid is expelled, the presence of as little as 0·1 c.c. of sulphuric acid causes slightly low results. The ferric complex is completely precipitated by ammonium sulphide; the aluminium complex is far more stable, not precipitated by ammonium sulphide, and destroyed only by evaporating with a large excess of strong nitric acid until fumes appear.

W. R. S.

Determination of the Rate of Solidification of Paraffin Wax. K. Fricke. (*Chem. Zeit.,* 1924, **48,** 624–625.)—A more definite indication of the value of paraffin wax for candle-making is obtained by measuring the rate of solidification than by determining the m.pt. A simple apparatus is designed for its measurement: 100 grms. of the wax are heated in a shallow tray to 65° C., then placed under a frame which holds vertically a rectangular iron rod which has a flat end and carries on the top a 2 kilo. weight. The rod is so arranged that it slides freely up and down. The time is noted from the attainment of 65° C. until, on applying the rod to the setting wax, it no longer makes any visible impression thereon. The temperature of the surrounding air should be from 20° to 22° C. Under these circumstances the best qualities of high m.pt. paraffin (54°/56° C.) should show a setting time of 50 to 60 minutes, and a medium quality from 60 to 70 minutes, whilst a common candle paraffin of m.pt. 50/52° C. takes 70 to 80 minutes.

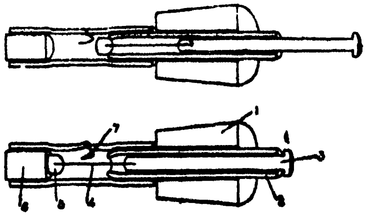
H. E. C.

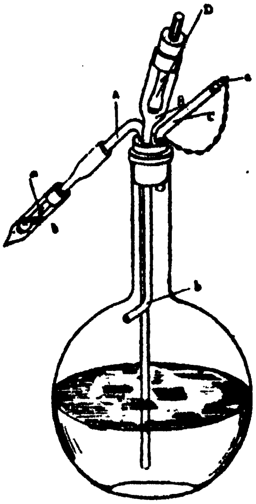
Measurement of the Viscosity of Heterogeneous Systems such as Paints. H. Wolff. (*Chem. Zeit.*, 1924, **48**, 647.)—In order to overcome the difficulties due to settlement in heterogeneous solutions, such as diluted paints or varnishes, the apparatus shown in the figure has been devised. The time in seconds is measured during which the weight *Gw* falls through a space corresponding to the distance between two marks on the spindle *S*; in falling the weight rotates the vane stirrer *F*, which is immersed in the paint in the pot *G*. The results obtained are quite empirical, but the measurements of viscosity in different dilutions in various solvents gives valuable information. An example is given showing that the viscosity of a paint when diluted with turpentine decreases as a simple linear function, but when the solvent is benzene or petroleum spirit there is a more rapid non-linear fall in viscosity. H. E. C.



Micro-analytical Technique. H. Häusler. (*Zeitsch. anal. Chem.*, 1924, **64**, 361–379.) **A. Benedetti-Pichler** (*ibid.*, 409–436).—The analytical method described makes use of a reaction vessel (small porcelain crucible, or beaker made from a test tube) and a filtering-tube, containing a minute plug of asbestos, through which the liquid is separated from the precipitate by suction while the vessel is at rest. The first paper gives directions for the manipulations and for the estimation of chlorine, nickel, mercury, nitric acid, calcium, barium, iron, copper, magnesium, and potassium. The tabulated results are very accurate. The method may also be used macro-analytically. The second paper contains diagrams of filtration apparatus, filtering tubes, beakers, etc., and directions for their manipulation. A detailed description is then given of the following separations: Silver from copper, calcium from magnesium, and potassium from sodium. W. R. S.

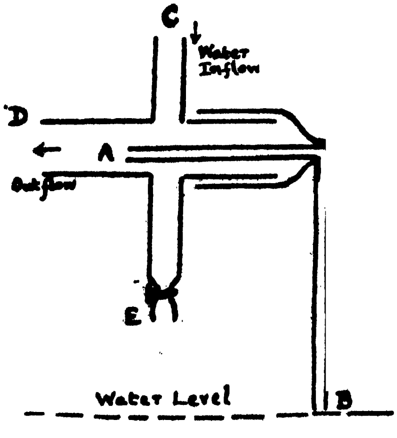
New Continuous-flow Wash-bottle. A. G. Nasini. (*Giorn. Chim. Ind. Appl.*, **1924**, **6**, 377.)—The part *a*, consisting of a glass tube, is joined to *A* by means of rubber tubing, and the Bunsen valve, which should be capable of acting in the air, prevents the tube *A* from becoming evacuated as a result of the external pressure. In the tube *B* is inserted, by means of a stopper, the part *D*, which is shown in detail separately; here the Bunsen valve has a wedge-shaped slit. When pressure is exerted on the glass tube 3, which is joined by a nickel wire to the ball 5, the pressure is transmitted to the glass plug 6. In this way the edges of the Bunsen valve are forced apart, the outside pressure being re-established in the flask; hence, the flow of liquid may be interrupted without removal of the bands from the jet or from the object being washed, as 3 may be pressed by the chin. If an increased flow of water is desired, the plug *c* is removed from *C* and the bottle inverted, air being drawn in through the valve *D* and the inclined tube *b*. The latter must not be immersed and if, in addition, the Bunsen valve is made of moderately thin rubber, the flow will be continuous. T. H. P.





Device for Maintaining a Constant Level in a Waterbath. R. Brooks. (*J. Chem. Soc.*, **1924**, **125**, 1546.)—The stream of water enters down *C* dividing between *D* and *E* (to the water bath) and together with the hydrostatic tension of the column of water from *D* to the sink draws air through *B* till water level is reached, and then water from the bath.

D. G. H.



Reviews.

A DICTIONARY OF APPLIED CHEMISTRY. By SIR EDWARD THORPE and Eminent Contributors. Volume V., Oxygen to Rye. Pp. 722. London: Longmans, Green & Co. 1924. Price 63s.

Sir Edward Thorpe is to be congratulated on his selection of contributors to this volume, whose names form an impressive list.

On examining the more important subjects treated, it is evident that each writer has an intimate knowledge of his subject at first hand, a feature which is by no means common to all publications dealing with the applications of chemistry.

The work under review is so well known to chemists that few comments are necessary. Each successive edition becomes more complete as regards subject matter than its predecessor, and the more modern advances in the branches dealt with in this volume are fully described. As in previous editions, an excellent account is given of the developments by Linde and Claude of the process for the manufacture of oxygen by the liquefaction of air, and the electrolytic processes in use at the present time are described in some detail. The relative costs of production by the "liquid air" and electrolytic processes are compared, and, in discussing the commercial applications of oxygen, the writer explains the advantage of the oxy-acetylene over the oxy-hydrogen flame in welding.

Under "Ozone" the various forms of ozonisers on the market are briefly described, and the efficiency, cost, and mode of application of ozone for the sterilisation of water supplies are discussed.

Full details are given in the section entitled "Electrical Precipitation" of the application of the Lodge-Cottrell process for recovery and utilisation of the waste products from flue gases.

Installations are described for the precipitation of acid mist from sulphuric acid concentrators, of precious metal dust from metallurgical works, and of dust from blast furnaces and cement kilns. This section will doubtless be of the greatest value to chemists and others engaged in the industries where the purification of waste gases can be advantageously applied.

A feature of the new editions the chapter on photosynthesis contributed by Professors Baly and Heilbron. In this chapter the writers discuss the fascinating problem of the course of the reactions occurring in the laboratories of the living plant cells, whereby the infinite varieties of complex carbohydrates, proteins, alkaloids, etc., are formed from carbon dioxide, water, and nitrate. An admirable historical introduction outlines the work of the pioneers in this field, an account being given of the experiments of Priestley, Ingen-Housz and de Saussure, which conclusively established the fixation of atmospheric carbon dioxide by the green part of the plant with the aid of sunlight, and the evolution of oxygen.

The observations of the above and subsequent workers led Baeyer to enunciate his celebrated hypothesis that formaldehyde is an intermediate product in the elaboration of sugars and starches by the living plant. The work of Willstätter in unravelling the complex constitution of chlorophyll and the accompanying pigments is next described, and Willstätter's views on the part played by chlorophyll in the assimilation process are outlined.

After the excellent summary mentioned above, the writers proceed to a formulation of their theory as to the mechanism of the process by which atmospheric carbon dioxide is converted to carbohydrates by the plant, and of how, by the addition of nitrates derived from the soil, the proteins and alkaloids occurring in the plant may be supposed to have been formed.

The theory enunciated is based on the assumption that formaldehyde is first formed by the reduction of carbon dioxide, and a general scheme is tabulated indicating the main lines along which the writers consider that the reactions proceed.

To explain the absence of formaldehyde from the leaf it is assumed that activated formaldehyde molecules have the constitution $\mathrm{H{-}\dot{C}{-}OH}$, and condense to form complex molecules without passing through the intermediate stage of formaldehyde in its stable form.

The reader will admire the ingenuity of the views propounded in this very stimulating article, and all students of the chemistry of plant life will eagerly await the results of the experiments now being carried out at Liverpool. Special attention might be well drawn to "Radiology," which is well to date in both theory and practical applications.

Whilst "Petroleum," as may be imagined, is a very ample section, with the various commercial tests described in detail, it is perhaps to be regretted that (probably for trade reasons) the methods of refining are hardly as modernised as could have been desired, but the information generally in the article is most valuable. In the space at the disposal of the reviewer it is impossible to do more than touch upon a few of the more salient features of this publication, and by far the greater part of the contents must be left without mention.

Throughout the book, errors and misprints are exceedingly rare, but on page 36, under Dental Porcelains, the word "Glucinum" is mis-spelt by the omission of "c," reading "Gluinum." The high standard attained in the previous volumes of this edition is fully maintained in Volume V., and the Dictionary can be unreservedly recommended as an authoritative source of information on applied chemistry.

GEO. R. THOMPSON.

THE COCOA AND CHOCOLATE INDUSTRY. A. W. KNAPP. Pp. 147. London: Sir Isaac Pitman & Sons, Ltd. 1923. Price 3s.

The author has aimed at producing a low-priced primer dealing with the whole subject of the cocoa and chocolate industries. He is to be congratulated on the amount of information, of interest alike to the general reader, the antiquarian, the scientific man, and the trade, which is compressed into the 147 pages of this little volume.

A clear distinction is drawn between the words "cocoa" (for the finished product) and "cacao" for the raw material, seeds, beans and butter, and it is greatly to be hoped that this distinction may soon become universal, thus obviating any confusion with coconut products.

The space devoted to the history of cocoa and chocolate will probably appear unduly generous to the average reader, but the appeal this aspect of the subject makes to the author seems irresistible. The account of the various factors influencing the growth of the cacao tree, the harvesting and treatment of the beans, and particularly the various manufacturing processes the beans are subsequently passed through, are clearly and concisely set out, although there are a few interludes written in the discursive and lighter vein beloved of the author.

The analytical data include the composition of cocoa essence, soluble cocoa, and cocoa shell, and the equivalent food units. No figures, however, are included for cacao butter, and although a short section is devoted to this product, it is not mentioned in the index.

The chapter on cost of production is most instructive, but the book as a whole is to be regarded by the analyst as more of a luxury than a necessity.

D. G. HEWER.

THE PLANT ALKALOIDS. By T. A. HENRY, D.Sc. Second Edition. Pp. viii.+ 456. London: J. & A. Churchill. 1924. Price 25s. net.

This, the second edition of Henry's "Plant Alkaloids," is a comprehensive book brought thoroughly up-to-date, and there has been enough important work on the chemistry of alkaloids, completed since the first edition was published ten years ago, to call for a new edition.

It is in all respects a very admirable production, complete, readable, concise, and well printed, with clear formulæ.

The chemistry of plant alkaloids is a very illuminating and fascinating part of our science. The unravelling of the constitution of the complex structure of alkaloids has occupied the attention of many of the world's best chemists, and the story of their achievements, as brought together in this book in readable condensed form, is worthy of the attention of all who are interested in organic chemistry.

The arguments based on experimental evidence by which the various constitutional formulæ have come to receive acceptance, make the book excellent reading to the student, to the research worker, and to the specialist.

The alkaloids are roughly classified according to the principal nitrogen-containing nucleus. This forms a convenient basis, and the best available in the present state of our knowledge, but is not of far-reaching significance. Such a book is helpful to workers in this field by laying bare the many gaps in our knowledge, and paving the way to a more complete classification and correlation of chemical constitution, biological occurrence and pharmacological action.

Each subject is dealt with in a historical manner, the principal physical and chemical properties of the bases and their salts are described, and there is given an account of the more important methods of isolation and analysis.

The pharmacological action of the successive groups is discussed, and in this respect more than in any other the gaps in our knowledge are conspicuous.

Ample references to original contributions are given at the foot of each page, and these greatly add to the value of the book.

Reading the book, one cannot fail to be impressed by the outstanding contributions of English chemists during the past twenty years, particularly those by what may be termed the Manchester school, Perkin, Pyman and Robinson.

FRANCIS H. CARR.

THE CHEMISTRY OF THE INORGANIC COMPLEX COMPOUNDS. AN INTRODUCTION TO WERNER'S COORDINATION THEORY. R. SCHWARZ. Authorised Translation by L. W. BASS, Ph.D. Pp. x.+82. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. Price 8s. 6d. net.

This little work supplies the long-felt need of a succinct account in the English language of Werner's theory. The translation, like the German original, presents "a synopsis of the Chemistry of the Inorganic Complex Compounds with as few details as possible." After an introductory chapter on valency, an *exposé* of the theory is given in Chapter II., followed by a substantial chapter on the isomerism of the complex compounds.

The translator rightly states that no English systematic nomenclature for the coordination compounds has yet been developed. However, committees appointed by the *Union Internationale de la Chimie Pure et Appliquée* are now engaged in elaborating a uniform terminology. It is to be hoped that their labours will soon bear fruit, and the nomenclature recommended by them be adopted by the whole profession. At the present time, the majority of British chemists conform, for the sake of uniformity, to the nomenclature used in the *Journal of the Chemical Society*, by referring to the latest Decennial Index; the terminology applied to inorganic complex compounds is an adaptation of Werner's German nomenclature, and appears to follow the rules given in Mellor's *Modern Inorganic Chemistry* (1917, p. 649). These the writer may be permitted to quote, as they are the only ones he could find in the English literature, and, moreover, they appear to be little known.

"Werner's system of naming the metal-ammonia compounds has been almost universally adopted. The constituents of the complex are taken first, and, of these, the acid radicles with the suffix "o" come first; then follow any groups which behave like ammonia, *e.g.* H_2O is called "aquo"; NO_2, "nitrito" or "nitro"; NO_3, "nitrato"; CO_3, "carbonato"; SO_3, "sulphito," etc., and lastly, preceding the metal itself, the ammonia molecules are designated "ammines," and spelt with a double "m" to distinguish the word from the "amines" or substituted ammonias. The prefixes di, tri, . . . indicate the number of each. The whole is written as one word." (Example: *chloropentamminechromic bromoiridate.*)

A somewhat similar system is applied in Roscoe and Schorlemmer's *Treatise*, as well as in the *Textbook* published under the editorship of Dr. Newton Friend. The writer regrets to note that Dr. Mellor, in compiling his great *Comprehensive Treatise*, is not adhering to the rules quoted from his earlier book, to judge from the term "*aurous dodeca-ammino-chloride*," and similar ones, in Volume III. The nomenclature outlined in the present translation is substantially the same as Mellor's older one, but the names of the coordinated radicals are separated from one another and from that of the central atom by hyphens. In addition, the valency of the central atom is shown by an ending: thus, $[Pt(NH_3)_3Cl_3]Cl$ is, *trichloro-triammine-platé chloride*, "*é*" indicating quadrivalency. The reviewer would like to see a substitute for the accented "*é*," which is un-English and not found on the typewriter keyboard.

In Volume IX., Part I., of his *Textbook*, Dr. Friend departs from Werner's nomenclature in applying the name "*ammoniate*" to the ammonia complexes of the bivalent metals, thus: $CoCl_2.6NH_3$, *cobaltous chloride hexammoniate*, instead of $[Co(NH_3)_6]Cl_2$, *hexamminecobaltous chloride*. The term brings out the analogy with $CoCl_2.6H_2O$, *cobaltous chloride hexahydrate*, but would lead to the inference that the above compound differs from *hexamminecobaltic chloride*, $[Co(NH_3)_6]Cl_3$ (luteo-salt), in the mode of linking of the ammonia molecules. As a matter of fact, whilst the cobaltous compound gives up its ammonia to the most dilute acids, the luteo-salt is remarkably stable. The results of the work of Biltz and his students, who are investigating the constitutional affinities between the two types of compounds, will, no doubt, have a decisive bearing on their terminology.

W. R. SCHOELLER.

BOILER CHEMISTRY AND FEED WATER SUPPLIES. By J. H. PAUL, B.Sc., F.I.C. 2nd Edition. Pp. ix.+252. London: Longmans, Green & Co. 1924. Price 14s. net.

This now well known book has deservedly reached a second edition. The text has been revised, and a number of fresh analyses of waters from various parts of the world have been added.

The subject matter is dealt with in 17 chapters, of which the first two are introductory—describing the origin of natural waters and the changes in their constituents which take place when passing through the soil, under the influence of chemical, biological and mechanical action. The author then attempts the difficult task of conveying, in popular language, for the benefit of non-scientific readers, the meaning of such terms as acids, bases and salts, of indicators, and of the nature of the changes which occur when salts are brought into solution in water.

The more technical portion of the work begins with the consideration of the normal constituents of natural waters, their solubility and the influence on them of high temperature when dry and in solution. The composition of boiler deposits and scales is illustrated with numerous analyses which shew the conditions under which such deposits had been produced, after which their prevention and cure are discussed. The consideration of the concentrated solutions remaining in the boiler naturally leads up to the important question of corrosion and its causes, and the special effects of carbon dioxide and alkaline carbonates under the influence of heat and pressure.

To complete this summary, the book deals with the condensed steam, if returned to the boiler, the removal of acidity and of oil, and, finally, with the problems arising in connection with the super-heater, with the cause and cure of priming, and with the damage to the external surfaces of boiler plates and tubes from the volatile constituents of the fuel.

The treatment of the subject is very complete and, while the practical side of boiler management is never lost sight of, the theoretical aspects of the problems involved are treated with an amount of detail which the growing knowledge of the subject now demands. The full story is yet to seek, but the mass of observations that have now been accumulated is gradually eliminating many of the early theories, and Mr. Paul's book has the merit of making a clear path through the maze of conflicting speculations.

The book is intended for the engineer as well as for the chemist, and this has necessitated the use of more or less popular phraseology and the intrusion of a certain amount of elementary chemistry which, from the point of view of the chemist, has involved some want of accuracy and precision. Such subjects as those dealt with in Chapter II (Acids, Bases and Salts), involving the use of indicators, and also the various theories of corrosion, cannot satisfactorily be explained without the ionic theory. The same remark applies to the tables of analyses of boiler waters, which might well mislead those who are not chemists into thinking that the salts are really combined in solution in the manner recorded, instead of these tables being merely conventional and convenient methods of stating the results.

Readers who are chemists certainly have a grievance against the author for providing no references to the literature of the subject. He states that "the principle of giving authorities is a subterfuge for passing on to others a responsibility which he has not the courage to assume on his own," but it is unreasonable to expect that all the statements in a book of this sort are the direct result of the writer's own work or observation. If these statements have any bearing on matters still in the controversial stage, the desire for the original sources of information is eminently reasonable, however carefully verified may be the facts.

The absence of these references makes it difficult to feel certain that the author himself has fully explored the literature of his subject. Certainly, some of the published work in connection with the effects of high pressure and temperature on water in boilers, on the action of dilute alkaline solutions on iron, and on the solubilities of some of the scale-forming salts appears to have had rather scant notice. The same may be said as to the various devices for the removal of oil; and the processes for the de-gassing of waters have been overlooked altogether.

These, however, are relatively small defects in a book which is a mine of original information, is fertile in suggestion, and eminently practical in its scope.

CECIL H. CRIBB.

CHEMICAL ENCYCLOPÆDIA. By C. T. KINGZETT, F.I.C. 3rd Edition. Pp x.+606. London: Baillière, Tindall & Cox. 1924. Price 30s. net.

The author is to be congratulated on the publication of the third edition of his book so soon after the second, for it is the best proof that the very large amount of work he has put into it has been appreciated by the particular class of readers he originally had in mind.

It will be noticed that whereas the former editions bore the title of "The Popular Chemical Dictionary," the limiting adjective has now been dropped, and the work has become a chemical encyclopædia.

Referring to this change in his preface, the author observes that the original title implied that his dictionary was not strictly scientific in character or accurate in detail, but it is open to question whether the more ambitious title is not likely to meet with more criticism from another direction. To be of use as an encyclopædia for the professional chemists mentioned in the preface, the work should give copious references to original papers and English abstracts of them, so that it could serve as a general subject-index in a much handier form than the bulky chemical dictionaries. If the author will adopt this suggestion, he will make a greater appeal than he can hope to do at present to practising chemists. As yet, the book must still be regarded mainly from the popular point of view—that is to say, as a readable digest of chemistry best suited to those who know but little of the science.

If this limitation is accepted, there is much to praise. The definitions are, as a rule, concise, yet clear, and the account of recent advances in all branches of chemistry is up-to-date and given in an interesting manner.

In the review of the last edition (ANALYST, 1922, **47**, 143) attention was directed to certain errors; these have now been corrected, but a few minor points call for correction in the next edition. On p. 273 the name of the Dutch chemist is given as "Wij" instead of "Wijs." It is not correct, without qualification as to the class of oil, to say (p. 213) that "the drying properties of oils are almost in direct ratio of their iodine values"; nor is it sufficient to say that "the acetyl value shows the amount of oxy-acids present," for it may show many other things. The statement about the use of graphite at Borrowdale (p. 86) is misleading, for the graphite used there is now imported from other places.

No man can hope to have a practical knowledge of all the subjects included in a chemical dictionary, and instances such as these only go to show the ad- ability of having the proof sheets revised by specialists in the various subjects.

The publishers have done their share of the work well, for the printing is ellent, and the binding and general style make the book very attractive in ance.

EDITOR.

Milk Products Sub-Committee.

UNIFORMITY OF METHODS OF ANALYSIS.

The above Committee will be glad to receive from Chemists particularly concerned with the analysis of Condensed Milk (sweetened and unsweetened), and of Dried Milk, details of unpublished methods, or variations in established methods, which they have found to be of service in their laboratories in such analysis.

Communications should be sent, if possible not later than the 6th December next, to the Hon. Secretary of this Committee,

MR. E. B. HUGHES, M.SC., F.I.C.,
7, Sumburgh Road, Clapham Common,
London, S.W. 12.

Publications Received.

ALLEN'S COMMERCIAL ORGANIC ANALYSIS. 5th Edition. Vol. II. (Fixed Oils, Fats and Waxes). J. & A. Churchill. 1924. Price 36s. net.

CHEMISTRY IN THE TWENTIETH CENTURY. Edited by E. F. Armstrong. E. Benn, Ltd. Price 15s. net.

QUANTITATIVE ORGANIC MICRO-ANALYSES. By F. Pregl. Translated by E. Fyleman. J. & A. Churchill. 1924. Price 12s. 6d. net.

VOLUMETRIC ANALYSIS. By Francis Sutton. 11th Edition. J. & A. Churchill. 1924. Price 35s. net.

THE EXTRA PHARMACOPOEIA. Vol. I. 18th Edition. Revised by W. H. Martindale and W. Wynn Westcott. H. K. Lewis & Co. 1924. Price 27s. net.

THE CONSTITUENTS OF COAL TAR. By P. E. Spielmann. Longmans & Co. 1924. Price 12s. 6d. net.

A SYSTEM OF PHYSICAL CHEMISTRY. By W. C. McLewis. Vol. III. (Quantum Theory). Longmans & Co. 1924. Price 15s. net.

L'ORIGINE TOURBILLONNAIRE DE L'ATOM ET LES CONSEQUENCES. Par J. Varin D'Ainvelle. Paris: Gauthier-Villars et Cie. 1924. Price 20 fr.

CHART OF THE CHEMICAL REACTIONS OF THE COMMON ELEMENTS. J. A. Timan. Chapman & Hall. 1924. Price 10s. net.

ERRATUM.

The title of Mr. Poucher's book reviewed in the October issue (p. 500) sh have read "Perfumes and Cosmetics with especial reference to synthetics."

PLEASE NOTICE.

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DECEMBER, 1924. Vol. XLIX., No. 585

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held in the Chemical Society's Rooms, Burlington House, on Wednesday, November 5, 1924. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. A. C. Barnes, B.Sc., F.I.C., J. J. Fox, D.Sc. (Lond.), F.I.C., I. C. Hay, and H. R. Jonson, M.Sc. (Liv.), A.I.C.

Certificates were read for the second time in favour of Messrs. A. C. Brooks, A.R.C.Sc.I., A.I.C., W. Donovan, M.Sc. (New Zealand), A. G. Flower, B.Sc. (Lond.), G. Hollingsworth, F.I.C., G. M. Norman, B.Sc. (Lond.), A.R.C.S., F.I.C., C. E. Sage, F.I.C., P. F. Spendlove, B.Sc., A.R.C.S., A.I.C., C. W. Spiers, M.Sc. (Bristol), A.I.C., L. H. Trace, B.Sc. (Lond.), A.I.C., and J. R. Walmsley, A.M.S.T., F.I.C.

Messrs. J. E. Nyrop and C. P. Thorpe were elected members of the Society.

The following papers were read:—"On certain new Methods for the Estimation of Arsenic and its Occurrence in Fish and Urine," by H. E. Cox, M.Sc., Ph.D., F.I.C.; "The Estimation of Cadmium in Brass," by A. T. Etheridge, M.B.E., B.Sc., F.I.C.; "The Hoyberg Method of Milk and Cream Testing," by N. C. Wright, B.A., and J. Golding, D.S.O., F.I.C.; and "An Apparatus for the Catalytic Dehydrogenation of Alcohols," by S. G. Willimott, Ph.D., B.Sc.

Deaths.

We deeply regret to have to record the deaths of the following Members of our Society:—

Sir Charles Cameron.
Dr. Robert Hellon.

Application of "Formol Titration" to the Kjeldahl Method of Estimating Nitrogen.

By W. S. SHAW, M.Sc., A.I.C.

(*Read at the Meeting, October* 1, 1924.)

If, in the Kjeldahl process, some means could be devised of estimating the ammonia formed which would render distillation unnecessary, considerable time and attention would be saved. It has been shown by Nanji and Shaw (ANALYST, 1923, **48**, 571) that the hypobromite method of estimating ammonia is unsuitable, since more than one reaction takes place when ammonia and hypobromite are brought together. The so-called "formol titration" method then suggested itself, and experiments were made to determine its accuracy and to devise a suitable modification of the method.

The "formol titration" method was devised by Sörensen (*Comptes rend.*, Carlsberg, 1907, **7**, I.; *Biochem. Zeitsch.*, 1907, 7, 45), and was applied by him to the measurement of the velocity of the hydrolysis of proteins by different enzymes. The reaction in the case of ammonium salts proceeds in accordance with the equation:

$$2(NH_4)_2SO_4 + 6HCHO = (CH_2)_6N_4 + 6H_2O + 2H_2SO_4.$$

Thus with ammonium salts, neutral hexamethylene tetramine is formed, with the liberation of acid equivalent to the nitrogen present as ammonium salts.

Malfatti (*Zeitsch. anal. Chem.*, 1908, **47**, 273) and Ronchese (*Bull. Soc. Chim.*, 1908, **3**, 840) obtained satisfactory results in the estimation of ammoniacal nitrogen by the "formol titration" method; but Van Bers (*Chem. Weekblad*, 1917, **14**, 968) found that the direct titration of the liberated acid gave unsatisfactory results. Kolthoff (*Pharm. Weekblad*, 1921, **58**, 1463) claimed, however, that accurate results could be obtained by the direct titration of the acid, provided that a sufficient excess of formaldehyde were used and the mixture allowed to stand for a minute before titration.

In order to test the accuracy of the method, and especially its application to the estimation of nitrogen in ammonium sulphate (the form in which it is present after the Kjeldahl digestion) experiments were made with a standard ammonium sulphate solution.

I. THE INDICATOR.—It is apparent that the indicator most convenient for use in the titration should be one whose colour changes should be definite and well marked, even at a low concentration of hydroxyl or hydrogen ions. The most sensitive indicator for acid and alkali in ordinary use is methyl red, as it is not affected by carbon dioxide, but it was found difficult to recognise the change to a distinct and definite orange coloration, and its use was discarded. Similarly with methyl orange.

The indicator finally adopted was phenolphthalein, and, in order to obtain stability at the end-point, the solutions were left slightly acid before the "exact" neutralisation and were boiled vigorously to expel carbon dioxide; they were then cooled before continuing the estimation. When this was done, no trouble was experienced in obtaining the required permanent tint.

II. NEUTRALISATION OF THE FORMALDEHYDE SOLUTION.—All previous workers on the "formol titration" followed the original method of Sörensen, and neutralised the formaldehyde with sodium hydroxide before use, phenolphthalein being used as indicator. The difficulty in the neutralisation of the formaldehyde lies in the fact that the "exact" neutral point cannot be obtained with a sufficient degree of certainty. The only method possible is to add alkali in the presence of phenolphthalein until a definite, slightly alkaline reaction is obtained. This seemed a possible source of error. Further, it was found that the degree of alkalinity obtained was very unstable owing to the high tendency of an alkaline solution of formaldehyde to become acid.

In order to overcome this difficulty of the neutralisation of the formaldehyde and escape the possible errors introduced by it, another method was adopted.

A solution of commercial formaldehyde (formalin) has usually a comparatively large free acidity, which may be determined by diluting 5 c.c. with about two or three times its bulk of water (previously boiled to expel carbon dioxide), and titrating the acidity with 0·1 *N* sodium hydroxide solution in the presence of about 3 to 6 drops of a 1 per cent. solution of phenolphthalein. Correction for the acidity of the formaldehyde solution used in the estimation can then be made, and by subtraction of this amount from the total titre obtained after the addition of the formaldehyde to the ammoniacal solution, a measure of the nitrogen is obtained. An advantage of this method is that the acidity of the formaldehyde solution remains constant during a considerable period, thus making only one estimation of the acidity necessary.

III. NEUTRALISATION OF AMMONIUM SULPHATE SOLUTION.—In dealing with the application of the "formol titration" to ammonium salts in general the type of the acid radicle of the salt must be borne in mind. It cannot be assumed that the salt of ammonia and a weak acid, such as acetic acid, would have a neutral reaction to indicators, for, by the ordinary laws of ionisation in solution, this is not so. Thus it is necessary in applying the method to ammonium salts, especially in the case of weak organic acid salts, that the solution be neutralised in the presence of phenolphthalein.

The actual neutralisation-point with phenolphthalein cannot theoretically be obtained, as the solution being titrated is always either colourless (hence acidic) or pink (hence alkaline). The actual tint of pink obtained in the estimation of the liberated acid by neutralisation is immaterial, provided it is not too remote from the neutral point, as a correction can be made for this. The main object is to obtain an end-point with a coloration which can be reproduced with ease and certainty. The best means of doing this is as follows:—Neutralise very carefully until the faint change from colourless to pink has taken place; add *one* extra drop of alkali, when a definite and clear pink colour will be obtained. After the addition of the formaldehyde solution the same procedure should be carried out in the neutralisation of the liberated acid, and the same tint will then be obtained. Thus any error which may be introduced by the varying behaviour of ammonium salts in solution may be overcome.

As the author was particularly concerned with the application of the "formol titration" to the Kjeldahl method of estimating nitrogen, experiments were carried out with an ammonium sulphate solution, under the conditions already given.

Five or ten c.c. of an ammonium sulphate solution containing 1 grm. per 200 c.c. are pipetted into a flask, and 5 drops of a 1 per cent. phenolphthalein solution added as indicator. No preliminary neutralisation of the ammonium solution is necessary, as it is already slightly acid. The contents of the flask are boiled vigorously to expel carbon dioxide, and the flask then cooled rapidly under cold running water. To this solution 0·1 *N* sodium hydroxide solution is added until the faint change to pink is noted; one extra drop of 0·1 *N* sodium hydroxide solution is added to produce a definite pink colour. The acidity of a formaldehyde solution is then determined as already described, 5 c.c. of this formaldehyde solution are added, and the mixture allowed to stand a few minutes. The liberated acid is titrated with 0·1 *N* sodium hydroxide solution, and the titre remaining after the deduction of the acidity due to the 5 c.c. of formaldehyde solution then affords a measure of the nitrogen present in the ammonium sulphate solution.

Following this procedure, a number of estimations were carried out on varying quantities of the ammonium sulphate solution, and the results only varied very slightly from the theoretical, usually within the magnitude of about ±0·03 to ±0·1 c.c. of 0·1 *N* sodium hydroxide solution (a deviation equal to approximately 0·07 mgrm. of nitrogen).

For comparison between the value obtained by the "formol titration" and by the distillation of the ammonia, 25 c.c. of the ammonium sulphate solution were distilled with an excess of sodium hydroxide. The result obtained, calculated as a percentage of nitrogen, showed a deviation of 0·19 per cent. from the value obtained by the "formol titration." The agreement is still closer if 0·05 *N* alkali is used.*

APPLICATION TO THE KJELDAHL METHOD.—The consistency and accuracy of the "formol titration" method of estimating nitrogen having been established, attempts were made to apply the method to the estimation of nitrogen in substances in which it can be estimated by the Kjeldahl method. It was recognised at the outset that two new factors would present themselves, namely (1) the presence of a comparatively large amount of electrolyte due to the neutralisation of the sulphuric acid and also of the potassium bisulphate added to raise the boiling point of the sulphuric acid with 40 per cent. sodium hydroxide solution; and (2) the presence of copper sulphate used as a catalyst in the digestion.

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*Prof: A. R. Ling attributes the discrepancy to the presence of carbon dioxide in the sodium hydroxide.—EDITOR.

In order to test the influence of sodium sulphate 10 c.c. of the standard ammonium sulphate solution were saturated with sodium sulphate and used for the estimation in the usual manner, but no deviation from the original results was obtained.

The copper sulphate present has a slight, though not material influence on the results of the titration. In alkaline solution a blue colour results, due to the formation of compounds with copper and alkali hydroxide and carbonates; the copper sulphate added, however, is only a very small quantity, and the alkalinity being also very slight, in the place of a blue coloration a slight turbidity results as the titration approaches the neutral point. The turbidity does not interfere with the estimation, since it is the same in the first neutralisation, *i.e.* before the addition of formaldehyde, as in the second neutralisation, so that, provided the tint, due to the phenolphthalein and the turbidity, is matched after each neutralisation, no error in the results is involved. Any difficulty in matching the tints may be avoided by adding two extra drops of alkali instead of one, so as to produce a still more definite pink coloration.

All the conditions present in the ordinary Kjeldahl process now being satisfied, two forms of procedure were devised according to whether there was a large or small amount of the material available for analysis.

A. LARGE AMOUNT OF MATERIAL AVAILABLE.—From 0·5 to 1 grm. (depending on the percentage content of nitrogen) of the material is weighed out and transferred to a Kjeldahl flask, and 15 c.c. of concentrated sulphuric acid and a crystal of copper sulphate added. The mixture is heated gently over a low flame until charring begins; 5 grms. of fused potassium sulphate are added, and the contents are then heated strongly until complete decarbonisation has taken place. This usually takes between 30 and 40 minutes. To ensure complete digestion the heating is continued for at least one hour after the liquid has become transparent.

The liquid is allowed to cool, 50 c.c. of distilled water added, and the mixture is boiled to expel any sulphur dioxide. After cooling, a piece of red litmus paper is added. Then 40 per cent. sodium hydroxide solution is run in cautiously, care being taken that the solution is left just acid, and that it is kept cool by immersing the bulb of the Kjeldahl flask under running water. The 40 per cent. sodium hydroxide solution added always contains appreciable quantities of carbonate, with the result that there is much carbon dioxide in the liquid; this fact makes it absolutely essential to boil before proceeding with the "formol titration" (*vide supra*). The slightly acid solution is transferred to a 250 c.c. graduated flask and made up to the mark, and is now ready for the titration of the nitrogen.

The procedure of the titration is divided into three definite parts, *viz.* (i) Preliminary neutralisation; (ii) "Accurate" neutralisation; (iii) Neutralisation of the acid liberated on addition of the formaldehyde. Twenty-five c.c. of the ammoniacal solution are pipetted into a flask, and 5 drops of a 1 per cent. solution of phenolphthalein added. The preliminary neutralisation, consisting in the neutralisation of the acid remaining after the approximate removal of the sulphuric acid used in the digestion by 40 per cent. sodium hydroxide solution is now effected by adding approximately 0·1 *N* sodium hydroxide solution, drop by drop, until a definite alkalinity is obtained, the alkali being well washed from the sides of the containing vessel. The solution is now made just acid with 0·1 *N* sulphuric acid, and boiled vigorously to expel carbon dioxide. It is then cooled rapidly and treated with 0·1 *N* sodium hydroxide solution, drop by drop, until the change from colourless to pink is just apparent, after which one drop more is added, to produce a decided pink coloration. This is the "accurate" neutralisation and, if any other dilution of sodium hydroxide is used for the titration, this same dilution must be used in the "accurate" neutralisation. Five c.c. of a formaldehyde solution, the acidity of which has previously been determined, are added, and the mixture allowed to stand for a few minutes. The acid liberated is now titrated with 0·1 *N* sodium hydroxide solution to the first appearance of a colour, and on the addition of one drop further (or, if necessary, 2 drops), the original tint should be obtained. Correction is then made for the acidity of the formaldehyde solution, and the amount of nitrogen in mgrms. present in the 25 c.c. of the solution is calculated.

It is probable in some cases that the formaldehyde solution may not have been added in sufficient quantity to react quantitatively with the nitrogen present; under such conditions 5 c.c. more of the formaldehyde solution are added and if, after the deduction of the extra acidity due to the extra 5 c.c. of formaldehyde solution, there is no change in the titre representing the nitrogen, no further addition is necessary. Five c.c. of an undiluted 40 per cent. formaldehyde solution were found to be more than sufficient to react with 15 mgrms. of nitrogen.

It is advisable to make two or three estimations with 25 c.c. of the solution, and to take the mean of the readings.

The results obtained with a number of nitrogenous substances are shown in the following table:

TABLE I.

Substance.	By Formol Method Per Cent	By Distillation Method Per Cent	Difference
Ammonium sulphate	20·58	20·77	−0·19
Acetanilide	9·91	9·71	+0·21
Asparagine	18·16	18·45	−0·29
Amygdalin	2·46	2·51	−0·05
Alanine	15·40	15·56	−0·16
Peptone	13·93	14·19	−0·26
Quinine sulphate	5·22	5·07	+0·15
Succinamide	24·06	24·20	−0·14

B. SMALL QUANTITIES OF MATERIAL AVAILABLE.—In cases where quantities of only 0·05 to 0·1 grm. of material are available the digestion is carried out in a boiling tube, as employed in micro-Kjeldahl digestions. The material (0·05 grm.) is weighed out and transferred to a boiling tube, 7·5 c.c. of concentrated sulphuric acid and a small crystal of copper sulphate are added, and the tube heated over a micro-Bunsen burner. As soon as charring begins 0·5 grm. of fused potassium bisulphate is added, and the liquid heated until colourless. It is then diluted and boiled, cooled and approximately neutralised, transferred to an Erlenmeyer flask, and titrated by the "formol" method.

Estimations of nitrogen in small quantities of the substances mentioned in Table I. gave results which did not vary from those obtained previously. Great care must be taken in the neutralisations that no alkali adheres to the side of the vessel, as considerable error can be introduced in this manner.

APPLICATION TO THE MICRO-KJELDAHL METHOD.—O. Folin and C. J. Farmer (*J. Biol. Chem.*, **1912**, **11**, 493) were the first to estimate nitrogen (in urine) by a micro-Kjeldahl method. In the digestion of the urine they used a mixture of 83 per cent. syrupy phosphoric acid (5 volumes) and concentrated sulphuric acid (1 volume). This mixture was advantageous from the standpoint of digestion, but had the disadvantage of attacking and devitrifying the glass of the boiling tube. The estimation of the nitrogen in the digested liquid was made by direct nesslerisation.

The most recent contribution to the literature on micro-Kjeldahl work is that of Ling and Price (*J. Soc. Chem. Ind.*, 1923, **41**, 149T). These authors discarded the digestion method of Folin and Farmer and adopted a new method on similar lines to the digestion in the ordinary Kjeldahl method, the ammonia in the distillate being determined colorimetrically with Nessler's reagent.

Experiments were carried out in which the strength of the alkali used in the "formol titration" was lowered considerably, the strengths varying from 0·1 *N* to 0·02 *N*. Nitrogen was estimated in equal portions of the digested acetanilide solution remaining from the former analysis in Table I., 0·1 *N*, 0·05 *N*, 0·04 *N*, and 0·02 *N* sodium hydroxide solution being used successively. The procedure of the titration is identical with that already given, with the exception that the lower the concentration of alkali used, the greater the number of drops of phenolphthalein that must be used. The results obtained were reduced to terms of 0·1 *N* alkali for comparison, and only a very slight divergence, amounting to a fraction of a mgrm. of nitrogen, was noted.

TABLE II.

Acetanilide solution taken	Strength of alkali used	Equivalent of nitrogen in 0·1*N* NaOH	Theoretical equivalent 0·1 *N* NaOH	Variation
c c	*N*	c c	c c	c c
25	0·1	6·55	6·55	—
25	0·05	6·51	6·55	0·04
10	0·04	2·63	2·62	0·01
10	0·02	2·64	2·62	0·02

From these figures it is clear that 0·02 *N* alkali may be used with accuracy in the "formol" titration. It is also evident that the error made in the titration is equivalent to 0·02 c.c. of sodium hydroxide solution, indicating an error of 0·028 mgrm. of nitrogen.

Two series of experiments were made with a standard ammonium sulphate solution. In Series I., 5 c.c. of a formaldehyde solution, containing 25 c.c. of 40 per cent. formaldehyde diluted to 100 c.c. were used; and in Series II., 5 c.c. of a formaldehyde solution containing 20 c.c. of 40 per cent. formaldehyde. The results obtained are given in Table III.

TABLE III.

Series.	Nitrogen by "Formol" Micro-Kjeldahl method. Mgrm.	Nitrogen calculated from "formol" method. Mgrm.	Error. Mgrm.
1.	0·802	0·824	−0·022
1.	0·423	0·412	+0·011
1.	0·211	0·206	+0·005
11.	0·825	0·824	+0·001
11.	0·409	0·412	−0·003

The degree of accuracy obtainable with the solution of digested quinine sulphate (*supra*) is shown in Table IV.

TABLE IV.

Amount of nitrogen solution taken. c.c.	Nitrogen found. Mgrm.	Nitrogen calculated. Mgrm.	Error. Mgrm.
2	0·409	0·418	−0·009
1	0·204	0·209	−0·005

The digestion is carried out under the conditions of Ling and Price (*loc. cit.*). An accurately weighed amount of the substance, containing approximately 1 mgrm. of nitrogen, is introduced into a boiling tube of hard glass, together with 1 grm. of fused potassium sulphate and 0·02 grm. of anhydrous copper sulphate. Five c.c. (*not* 8 c.c) of concentrated sulphuric acid are added, together with two drops of a 2·5 per cent. platinum tetrachloride solution. A small funnel is placed in the mouth of the tube, and the contents are boiled gently until the liquid is colourless. The liquid is cooled, 10 c.c. of distilled water are added, and the solution boiled to expel sulphur dioxide.

The solution is again cooled and approximately neutralised with 40 per cent. sodium hydroxide solution, the amount added not exceeding 15 c.c., thus leaving 20 c.c. for the subsequent washing. The solution is now transferred to a 50 c.c. graduated flask, the boiling tube being well washed out. The nitrogen in an aliquot portion of the resulting liquid, say 10 c.c., is now estimated under the conditions necessary for the "formol titration" (*supra*).

When the nitrogenous material to be analysed is in the liquid form, as, for example, in the case of vaccines, a slightly modified digestion is necessary. If the 5 c.c. of concentrated sulphuric acid are added to the vaccine and heated, there is an inclination to excessive bumping, which must be avoided. To overcome this, 5 c.c. of the vaccine are pipetted into the boiling tube, 1 c.c. of concentrated sulphuric acid is added, and the tube immersed in a boiling water bath until a slight amount of charring occurs; the remainder of the 5 c.c. of concentrated acid is now added, and the digestion continued in the usual manner.

The author wishes to thank Professor A. R. Ling and Mr. D. R. Nanji for their advice and encouragement in the course of this work.

UNIVERSITY OF BIRMINGHAM,
DEPARTMENT OF THE BIOCHEMISTRY OF FERMENTATION.

The Pemberton-Neumann Method for the Estimation of Phosphorus.

BY MARION B. RICHARDS AND WILLIAM GODDEN.

(*Read at the Meeting, October* 1, 1924.)

THE authors have had occasion to use this method for the estimation of phosphorus in a large number of samples of urine and fæces, and at the outset, during a trial of the method, found certain discrepancies in the results if the conditions of experiment were varied to any marked extent. An examination of the literature showed that this fact was recorded by numerous workers, who had also investigated the cause. There was, however, no definite agreement as to the cause of the errors or as to the steps to be taken to avoid them. Accordingly it was decided to enquire further into the method, with a view to establishing a definite procedure which would give consistent results with varying amounts of material.

ORIGINAL METHOD.—The method, as originally outlined by Pemberton[1] for inorganic phosphates, is briefly as follows:—The phosphate is dissolved in nitric acid, the solution made just alkaline with ammonia, and 5 c.c. of nitric acid (sp. gr. 1·4) and 10 c.c. of a saturated solution of ammonium nitrate are added. Into this solution, when boiling, a slight excess of a cold aqueous solution of ammonium molybdate is run, and, as soon as the precipitate has settled, it is filtered off, washed with cold water and dissolved in a known volume of 0·5 *N* potassium hydroxide solution. The excess of alkali is then titrated back in the cold with 0·5 *N* sulphuric acid, without boiling off the ammonia, phenolphthalein being used as indicator. Pemberton gives the molecular ratio of P_2O_5 to KOH as 1:46, *i.e.* 1 c.c. of 0·5 *N* KOH is equivalent to 0·001543 grm. of P_2O_5.

Neumann[2] modifies this method, in that he precipitates in the presence of sulphuric and nitric acids, using 50 c.c. of a 50 per cent. solution of ammonium nitrate instead of 10 c.c. of a saturated solution, and he precipitates at 70°–80° C. Finally he boils his precipitate with an excess of 0·5 *N* sodium hydroxide solution to remove the ammonia and then titrates back the excess alkali, in the cold, with

0·5 *N* acid. He gives the molecular ratio P_2O_5 to NaOH as 1:56, *i.e.* 1 c.c. of 0·5 *N* NaOH is equivalent to 0·001268 grm. of P_2O_5.

COMPOSITION OF PRECIPITATE AND FACTORS.—It should be noted that both these authors cite Hundeshagen[3] as to the composition of the phosphomolybdate precipitate, but each takes a different formula without, apparently, having paid particular attention to the details given by Hundeshagen. This author found that the composition of the ammonium phosphomolybdate precipitate, when obtained under the most varied conditions, after washing with cold dilute nitric acid, and drying at 130°–150° C., in all cases corresponded in composition with the formula $(NH_4)_3PO_4$, $12MoO_3$. This was the case whether the precipitation took place in strongly or weakly acid solution, whether in the presence of nitric, hydrochloric or sulphuric acid, whether concentrated or dilute, in hot or cold solution, with phosphate or molybdic acid in excess, and whether other salts (*e.g.* of potassium) were present or not.

If precipitated in the presence of excess of nitric acid, washed with cold dilute nitric acid and dried in a desiccator over calcium chloride and potassium hydroxide to constant weight, the precipitate had the composition $(NH_4)_3PO_4$, $12MoO_3$, $2HNO_3$, H_2O. The acid and water were probably in loose combination and were quickly and completely removed by heating at 150° C.

Pemberton, in calculating his factor, took the formula without the two molecules of nitric acid and did not allow for the alkali necessary to replace the ammonia, as this was not removed by boiling. Neumann, on the other hand, took the formula with the two molecules of acid and allowed for the alkali necessary to replace the ammonia removed by boiling. Both of these workers washed their precipitate with water, Pemberton stating, contrary to Hundeshagen's findings, that the precipitate is not dissolved by the water even if one litre is used for the washing. Their two equations are respectively:—

$$\begin{aligned}2(NH_4)_3PO_4,\ 24MoO_3+46NaOH &= (NH_4)_4H_2P_2O_8\\ &\quad +(NH_4)_2MoO_4+23Na_2MoO_4+22H_2O\\ 2(NH_4)_3PO_4,\ 24MoO_3,\ 4HNO_3+56NaOH &= 24Na_2MoO_4\\ &\quad +2Na_2HPO_4+4NaNO_3+6NH_3+32H_2O.\end{aligned}$$

Gregersen[4] modified Neumann's method in so far as the treatment of the precipitate is concerned. After boiling with the 0·5 *N* acid he recommends the addition of a slight excess of the acid and second boiling to expel carbon dioxide, the solution being finally titrated back when cold with the 0·5 *N* sodium hydroxide solution. He apparently retains the factor, 1 c.c. of 0·5 *N* NaOH = 0·001268 grm. P_2O_5, as given by Neumann.

Heubner[5], and later Jodidi[6], examined Neumann's method and were in agreement that Neumann's factor is too low. They both recommend as the factor 1 c.c. of 0·5 *N* NaOH = 0·0057 grm. of P (or 0·001305 grm. of P_2O_5), basing their figure, not on any equation, but by checking it against pure phosphate solutions which had been analysed gravimetrically. Wardlaw[7] suggests that the factor needs to be varied with the amount of phosphorus present.

PHOSPHORUS IN BIOLOGICAL MATERIAL.—The method has in all cases been tested upon pure inorganic phosphates, but in the estimation of phosphorus in biological material the process for the conversion of the phosphorus into phosphoric acid and the subsequent preparation of the solution for the precipitation need to be considered. Neumann (*loc. cit.*) recommends that the material should be digested with 20 c.c. of a mixture of equal volumes of concentrated sulphuric and nitric acids, more of this mixture being added from time to time if necessary. This solution he simply dilutes and then proceeds as described. Gregersen (*loc. cit.*) uses 20 c.c. of the acid mixture at the commencement, as described by Neumann, but, if further acid is necessary, adds nitric acid alone, as had been previously recommended by Plimmer and Bayliss[8]. Further, he does not neutralise after dilution before the precipitation. The procedure we have adopted, as being more in accord with Hundeshagen's results, is to carry out the oxidation as described by Gregersen, thereby limiting the amount of sulphuric acid to 10 c.c. Subsequently, when oxidation is complete, the solution is diluted to 200 c.c., made just alkaline with ammonia and then just acid with nitric acid. Thirty c.c. of a 50 per cent. solution of ammonium nitrate are now added, thus bringing the concentration of the ammonium nitrate in the total bulk of solution to about 7 per cent. (*cf.* Hundeshagen). The precipitation is carried out at 70 to 75° C., a solution of ammonium molybdate, prepared, as described by Mathews[9], from ammonium molybdate and not from molybdic acid, being used. We do not wash the precipitate with water only, but twice with 10 per cent. nitric acid to remove any excess of ammonium molybdate, three to four times with 2 per cent. ammonium nitrate solution to remove the acid, and then twice with cold water to remove the ammonium nitrate. The reason for this will be discussed later. The filtration and washing are carried out with the aid of suction on a Hirsch funnel, a small disc of hardened filter paper being used. By this means washing is more rapid and thorough, and the amount of filter paper present during the boiling with the alkali is reduced to a minimum.

EXPERIMENTAL.—The possible sources of error in the method, which were examined, were:—(1) Variation in the volume of semi-normal alkali used to dissolve the precipitate by Neumann's method; (2) failure to boil with excess of acid before final titration; (3) variation in the amount of phosphate present keeping the volumes of precipitation reagent constant; and (4) variation in the volume of ammonium molybdate used for the precipitation. Finally the correct factor to be used in the calculation had to be considered in the light of these results.

(1) *Variation in the volume of standard alkali used to dissolve the precipitate.*—When boiling the precipitate with alkali by Neumann's method, taking no particular precautions as to the amount of the alkali in excess, it was found that duplicate results often showed wide variations. Accordingly a solution of pure sodium phosphate was prepared which, on analysis, both gravimetrically and by the uranium acetate method, was found to contain 0·02575 grm. of P_2O_5 in 25 c.c. Of this solution, 25 c.c. portions were pipetted out, and the precipitate obtained was filtered off and washed as described above. This precipitate was dissolved

in varying amounts of 0·5 *N* sodium hydroxide solution, and, after the liquid had been boiled for twenty minutes and cooled, the excess of alkali was titrated.

TABLE I.

Vol. of 0·5 *N* NaOH used to dissolve precipitate. c.c.	Vol. of 0·5 *N* H_2SO_4 required for back titration. c.c.	P_2O_5 in 25 c.c. of solution, calculated with Neumann's factor. Grm.
20·00	0·15	0·02517
20·00	0·15	
21·00	0·95	0·02542
21·00	0·95	
22·00	1·70	0·02574
22·00	1·70	
24·00	3·20	0·02637
24·00	3·20	
26·00	5·00	0·02663
26·00	5·00	
28·00	6·85	0·02681
28·00	6·75	0·02694

Thus the amount of alkali used to dissolve the precipitate, under Neumann's conditions, played a considerable part in determining the result, which was by no means constant when the amount of alkali varied. The most accurate result was obtained when 1 to 2 c.c. in excess of alkali was used. (Neumann used 5 to 6 c.c. in excess.)

(2) and (3) *Failure to boil with excess of acid and variations in the amount of phosphate present.*—For this purpose a solution of pure potassium dihydrogen phosphate was prepared and analysed gravimetrically, (*a*) by weighing the phosphorus as $Mg_2P_2O_7$, and (*b*) as KPO_3.

50 c.c. of the solution contained by (*a*) 0·02589 P_2O_5.
50 c.c. of the solution contained by (*b*) 0·02596 P_2O_5.

The mean figure 0·02593 was taken.

Varying amounts of this solution were used, and the estimations carried out as usual, the end-point being obtained first under Neumann's conditions and then under Gregersen's conditions. The results were all calculated for 50 c.c. of the original solution and are given in Table II.

TABLE II.

	Neumann's conditions.		Gregersen's conditions.
Vol. of phosphate solution taken. c.c.	0·5 *N* alkali consumed. c.c.	Same calculated for 50 c.c. of solution. c.c.	0·5 *N* alkali used calculated for 50 c.c. c.c.
5	2·60	26·00	19·04
10	4·81	24·05	19·04
20	8·14	20·35	19·04
30	12·10	20·16	19·08
40	15·67	19·59	19·08
50	19·49	19·49	19·04

With Neumann's factor volume of 0·5 N alkali should have been 20·45 c.c.
„ proposed new „ „ „ „ „ „ „ „ 19·00 c.c.

It will at once be seen that concordant results are not obtained with varying amounts of phosphate, under Neumann's conditions, but that the agreement is excellent if Gregersen's modification of boiling with the excess of acid, prior to the final titration, is adopted. The question of the proposed new factor will be discussed later.

That this discrepancy between the results, under Neumann's conditions, is due to carbon dioxide absorbed by the alkali during the boiling and cooling, as stated by Gregersen, is readily shown by the following data. Ten c.c. portions of a solution of sodium phosphate were taken, and diluted to 200 c.c., and to each of them were added 5 c.c. or 10 c.c. of 0·5 N sodium hydroxide solution. The solutions were then boiled in a round-bottomed flask for twenty minutes and, after cooling, titrated back with 0·5 N sulphuric acid in the presence of phenolphthalein. An excess of acid was next added and, after further boiling for twenty minutes, the solutions were again cooled and titrated back with 0·5 N sodium hydroxide solution. The results are given in Table III.

TABLE III.

Before boiling with excess of acid.			After boiling with excess of acid.		
Vol. of 0·5 N NaOH added.	Vol. of 0·5 N H_2SO_4 required.	Difference.	Total Vol. of 0·5 N NaOH used.	Total Vol. of 0·5 N H_2SO_4 used.	Difference.
c.c.	c.c.	c.c.	c.c.	c.c.	c.c.
5	4·3	0·7	6·0	6·0	0·00
5	2·85	2·15	6·0	5·95	0·05
10	8·90	1·10	11·0	10·90	0·10
10	6·65	3·35	11·0	10·95	0·05
10	8·25	1·75	11·0	11·00	0·00

Had there been no absorption of carbon dioxide the figures in columns (1) and (2) should have been identical. They are different, however, and there is no regularity, owing probably to the different rate of boiling, time of cooling, and state of the atmosphere. After expelling the carbon dioxide, however, by boiling with excess of acid the agreement is, with one exception, excellent. Finally, when the experiment was repeated, with 10 c.c. of 0·5 N NaOH, with boiling and cooling under such conditions as to exclude carbon dioxide, the first titration required 10 c.c. of 0·5 N sulphuric acid, and boiling with excess of acid did not alter the agreement. (Jodidi recommends the use of a correction of 0·2 c.c., based on a series of blank analyses to compensate for the errors due to (*a*) impurities in the reagents, (*b*) carbon dioxide in the liquids to be titrated, and (*c*) acidity caused by the action of the boiling alkali on the filter paper (a 12½ cm. folded paper). In the light of the above figures such a correction is not permissible.)

(4) *Variation in the volume of molybdate solution used for the precipitation.*—A solution of disodium hydrogen phosphate, containing 0·01574 grm. of P_2O_5

in 25 c.c. was used. Separate 25 c.c. portions were treated with varying volumes of molybdate solution, as shown, but otherwise the procedure described above was followed, and the liquid was boiled with excess of acid before the final titration.

TABLE IV.

Vol. of molybdate used.	Vol. of 0·5 *N* NaOH consumed.	Vol. of 0·5 *N* NaOH calculated from.
c.c.	c.c.	
10	11·48	Factor 0·001365
20	11·50	11·53 c.c.
30	11·52	Factor 0·001268
50	11·65	12·41 c.c.

NOTE.—Ten c.c. of this molybdate solution are about 1 c.c. in excess of that required theoretically to precipitate all the phosphate which was present.

Within reasonable limits, therefore, the volume of the molybdate solution used for the precipitation does not interfere with the results.

FACTOR.—It will be noticed that in Tables II. and IV. a new factor (1 c.c. of 0·5 *N* NaOH ≡ 0·001365 grm. of P_2O_5) has been used, and this is the factor by means of which it is proposed all calculations should be made under the conditions laid down in this paper. It is based in the first place on the work of Hundeshagen (*loc. cit.*), and in the second place on our own experimental results. Hundeshagen has shown that, while the composition of the yellow precipitate, obtained in the presence of nitric acid and washed with dilute nitric acid, is $(NH_4)_3PO_4$, $12MoO_3$, $2HNO_3$, the nitric acid is very loosely combined, and may be removed (*a*) by heating at 130° C., and (*b*) by washing with neutral aqueous ammonium nitrate solution. He suggests that it is possible that this nitric acid may be replaced in the molecule by ammonium nitrate under such conditions. As, however, we follow the washing with the ammonium nitrate by washing with cold water, we are of the opinion that the resulting precipitate left on the filter paper has the composition $(NH_4)_3$, PO_4, $12MoO_3$. This is confirmed by the figures given in Tables II. and IV. Further confirmation is given by the following data. Three separate 50 c.c. portions of the solutions used for the experiments recorded in Table II. were treated with molybdate, and the precipitates filtered off and washed in the usual manner. In two cases the precipitates were heated at 150° C. before solution in the standard alkali, whilst the third precipitate was dissolved at once. The following results were obtained:

Duration of heating at 150° C.	Vol. of 0·5 *N* NaOH used up.	Vol. of 0·5 *N* NaOH calculated with factor 0·001365.
	c.c.	c.c.
2½ hours	19·00	—
4½ "	19·02	19·00
nil	19·02	—

The heating at 150° C. would certainly, according to Hundeshagen, remove any loosely-combined nitric acid and, as the heating in this manner has not affected the results, the precipitate left under the given conditions must be free from combined nitric acid. Thus the reaction with the alkali will be represented by—$2(NH_4)_3PO_4,24MoO_3 + 52NaOH = 24Na_2MoO_4 + 2Na_2HPO_4 + 28H_2O + 6NH_3$; the molecular ratio of P_2O_5 to NaOH is 1:52, and the factor 1 c.c. of 0·5 *N* NaOH = 0·001365 grm. of P_2O_5.

PROPOSED METHOD FOR BIOLOGICAL MATERIAL.—The following details are given for the estimation of total phosphorus in fresh fæces: From 1·5 to 2·0 grms. are weighed out and transferred to a 500 c.c. round-bottomed flask, 10 c.c. of concentrated sulphuric acid and 10 c.c. of concentrated nitric acid are added, and the mixture is digested over a low flame until brown fumes cease to be evolved and the flask is full of white fumes. The mixture is allowed to cool, a further 5 c.c. of nitric acid are added, and the digestion is continued until the liquid in the flask is quite clear and colourless. When cold, the solution is diluted to about 200 c.c., made just alkaline to litmus with concentrated ammonia, and then just acid with nitric acid. Thirty c.c. of a 50 per cent. solution of ammonium nitrate are added, the liquid is heated at 70 to 75° C., and 30 c.c. of the ammonium molybdate solution (to which 1·5 c.c. of concentrated nitric acid have been added) are run in, and the mixture is well shaken. The precipitate is allowed to settle until the liquid is cold (usually one hour is sufficient, but the result is not altered if it is left to stand overnight). It is filtered off, by suction, on a disc of hardened filter paper in a Hirsch funnel, and is washed twice with 10 per cent. nitric acid, three to four times with 2 per cent. ammonium nitrate and twice with cold water. The precipitate and filter paper are washed back into the precipitation flask with cold water and dissolved in a known volume of 0·5 *N* sodium hydroxide solution (about 1 c.c. in excess being used). The solution is diluted to about 250 c.c. and boiled for twenty minutes. While still warm it is titrated back with 0·5 *N* sulphuric acid, phenolphthalein being used as indicator, and an excess of 1 to 2 c.c. of acid is run in. Boiling is repeated for 15 minutes and, after cooling, the excess acid is accurately titrated with 0·5 *N* sodium hydroxide solution. The end point is taken as soon as the first definite pink tint is obtained, although the colour will be found to fade fairly rapidly. The total alkali used, less the total acid, gives the volume of alkali equivalent to the P_2O_5 in the original weight of fæces (1 c.c. of 0·5 *N* NaOH = 0·001365 grm. of P_2O_5). It is desirable that the actual amount of phosphoric anhydride present for precipitation should not exceed 22 mgrms., or there may be some difficulty in washing the precipitate completely free from nitric acid and ammonium nitrate.

Note.—The authors have found one Winchester of nitric acid out of a large consignment to contain very decided amounts of phosphoric anhydride. One lot of ammonium nitrate also contained 0·003 per cent. of P_2O_5. The impurity in the nitric acid was presumably due to faulty washing of the containing vessel prior to its being filled by the manufacturer.

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THE ROWETT RESEARCH INSTITUTE,
ABERDEEN.

The Estimation of Cadmium in Brass.*

BY A. T. ETHERIDGE, M.B.E., B.Sc., F.I.C.

(*Read at the Meeting, November* 5, 1924.)

CADMIUM is associated with zinc in zinc ores, and is therefore present in commercial zinc, and, consequently, in brass. The amount found in brass varies from a mere trace (less than 0·01 per cent.) up to 0·2 per cent., depending on the quality of the zinc used. The effect of cadmium on the properties of brass is not yet completely known. In particular cases specifications are laid down fixing a maximum amount permissible, usually 0·05 per cent.

Cadmium sulphide can be precipitated practically free from zinc from slightly acid solutions, but it is necessary to remove copper first. In the work described in this paper the electrolytic method, which has great advantages over chemical methods, was used. It is necessary to take at least 5 grms. of brass. If the copper is removed as thiocyanate, in addition to the difficulty of filtering and washing, it is almost certain that a large proportion of the cadmium will be occluded in the precipitate. If the copper is removed as metal by sodium hypophosphite it is impossible to adjust the solution afterwards for the precipitation of cadmium in presence of zinc, on account of the phosphoric acid formed. In the electrolytic method 5 grms. of brass can be easily handled and no disturbing compounds are left behind.

The method is as follows:—Five grms. of brass, preferably drillings or turnings (freed from iron particles by a magnet) are dissolved in a mixture of 80 c.c. of dilute sulphuric acid (1 vol. conc. acid to 3 vols. water) and 7 c.c. of nitric acid (sp. gr. 1·42) in a 400 c.c. beaker. When the brass is dissolved and the nitrous fumes have been expelled, 150 c.c. of water are added and the beaker is cooled in running water for about 1 hour. Any lead present is partly precipitated at this stage as lead sulphate. The precipitation is not quite complete owing to the slight solubility of lead sulphate in liquids containing small amounts of nitric acid. As it is important that there should be no suspended lead sulphate carried forward to the next stage, it is better in all cases to pass the liquid through a small close pulp filter, whether any lead is visible or not. This point is referred to again later. The filtrate is caught in a 400 c.c. squat beaker and with the washings its volume is about 200 c.c. The electrolysis is carried out in the manner described by the author in the paper on "Analysis of Copper-Tin Alloys" (ANALYST, 1924, 371). A rotating platinum gauze cathode and platinum foil anode are used. The initial current is 4 amps. After 30 minutes 0·5 grm. of urea is added to destroy nitrous acid. When the liquid becomes colourless the current is continued at 4 amps. for another 30 minutes, after which it is reduced by ½ amp. every 15 minutes. At each change 0·5 grm. of urea is added. At the end of the last 15 minutes at ½ amp. the beaker is lowered away from the cathode, which is washed from the wash-bottle jet at the same time. The anode is also washed and lifted out. Traces of lead will probably be observed, as peroxide, on the anode, due to the presence of nitric acid, in which lead sulphate is slightly soluble. The remainder of the lead thus held in solution is completely separated on the anode at this stage. It would not be safe, however, to assume that suspended lead sulphate could be quantitatively transferred to the anode in this manner; hence the necessity of filtering before electrolysis. There must be no lead in the liquid after electrolysis, as it would contaminate cadmium separated in subsequent stages.

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* Communication from the Research Dept., Woolwich.

The liquid left contains all the cadmium and zinc, and the following metals may also be present: Arsenic, antimony, tin, bismuth, iron, nickel, manganese. No cadmium is deposited on copper under the conditions given, but antimony and bismuth are partly precipitated, as shown by discoloration of the deposited copper. The next step is to remove all nitric acid by careful evaporation to incipient fuming. This operation requires several hours, and must be carefully watched to avoid spirting when zinc sulphate begins to separate out. The elimination of nitric acid is necessary for the subsequent adjustment of acidity for the next operation.

After cooling, 200 c.c. of cold water are added quickly, and the liquid is stirred to dissolve the sulphates. Over-heating must be avoided in case tin is present. (Admiralty brass contains 1 per cent. of tin.) The liquid is cooled and neutralised, as shown by litmus paper, with dilute ammonia (one vol. strong ammonia to one vol. water). The volume should be approximately judged (usually 300 c.c.); it is then adjusted to correct acidity for precipitation of cadmium by adding 7 c.c. of dilute sulphuric acid (1 vol. conc. acid to 3 vols. water) per 100 c.c.

FIRST ACID PRECIPITATION.—The liquid is cooled, hydrogen sulphide is bubbled through it in a rapid stream for 30 minutes, and the beaker is then set aside to stand over-night or for at least 12 hours. The cadmium sulphide is not usually visible at this stage unless it is large in amount (of course it may be obscured by tin). On standing, it gradually collects and falls to the bottom of the beaker. It is probably in a colloidal state at first, like arsenic sulphide. The failure to recognise the importance of long standing has probably led to recommendation of the use of trichloracetic acid instead of sulphuric acid; also to the practice of purposely allowing some zinc sulphide to form, and increasing the acidity afterwards. These variations may cause cadmium sulphide to collect more rapidly, but they have disadvantages. In any case, in dealing with minute precipitates, it is generally a mistake to attempt to filter too soon after precipitation. Sulphuric acid is quite satisfactory in this case if long standing is allowed.

The precipitate may contain, as well as cadmium sulphide, traces of copper (if directions for electrolysis are not properly followed), tin, arsenic, antimony, bismuth and zinc, but it is nearly free from iron, nickel and manganese. The precipitate is filtered on a paper pulp filter and washed with 1 per cent. ammonium nitrate solution. Washing with water tends to carry some precipitate through the pulp. If tin is present as a constituent, as in Admiralty brass, the bulk of the precipitate is large. In that case some zinc sulphide is certain to be in it, apart from the impossibility of washing all the zinc sulphate out. In the absence of tin the amount of zinc entangled is small, but it is always present at this stage.

Arsenic is probably partly removed as hydrogen arsenide during electrolysis, but the remainder is precipitated at this stage. Antimony and bismuth are partly deposited on the cathode, as mentioned above, and the remainder is precipitated at this stage. Iron, etc., are washed out (except traces), the amount left depending on the bulk of the precipitate.

FIRST ALKALINE PRECIPITATION.—The precipitate is dissolved in hot dilute (1:1) *aqua regia* (1 vol. nitric acid of sp. gr. 1·42, and 3 vols. hydrochloric acid of sp. gr. 1·2 and 4 vols. water) and the filtrate is caught in the original beaker. After boiling for a few minutes to decompose the sulphides and cooling to room temperature, it is made alkaline with a slight excess of dilute (1:1) ammonia solution, and a crystal of potassium cyanide is added in cases where traces of copper are suspected. Hydrogen sulphide is then passed through in a rapid stream for about 15 minutes. Tin is precipitated by ammonia but is re-dissolved after hydrogen sulphide has passed through for a short time. The precipitate is cadmium sulphide together with any zinc and bismuth in the first "acid precipitate," but is free from arsenic, antimony and tin, if these were only present as traces in the original alloy. If tin is a constituent of the alloy, traces of tin will still remain in the precipitate. Traces of iron are also usually to be found here. It is not necessary to allow this precipitate to stand for 12 hours; cadmium sulphide collects very quickly from an alkaline liquid and may be filtered after 2 hours' standing. It is washed, as before, with 1 per cent. ammonium nitrate solution.

SECOND ACID PRECIPITATION.—The precipitate is dissolved as before, the liquid is caught in a 200 c.c. tall narrow beaker, and after the addition ot 20 c.c. of dilute sulphuric acid, it is evaporated until fumes appear. After cooling and diluting to 80 c.c. it is neutralised with ammonia. The volume is judged (usually 100 c.c.) and the acidity adjusted, as before, by the addition of 7 c.c. of dilute sulphuric acid per 100 c.c. When cool, a rapid current of hydrogen sulphide is passed through for 30 minutes, and the liquid is set aside for at least 12 hours, as in all "acid precipitations."

The precipitate contains all the cadmium, with traces of tin, if tin was present originally as a constituent, with part of the original bismuth, which also follows the cadmium throughout after the electrolysis stage, but it is now nearly free from zinc and quite free from iron, nickel, etc.

SECOND "ALKALINE PRECIPITATION."—(If tin, arsenic and antimony are present as impurities only in the original alloy, this stage should be omitted.) The precipitate is filtered off, dissolved, etc., exactly as in the first alkaline precipitation (with omission of potassium cyanide, which is unnecessary at this stage). This eliminates the remaining traces of tin in cases where tin is present as a constituent.

THIRD "ACID PRECIPITATION."—This is exactly the same as the second acid precipitation. This stage eliminates the remainder of the zinc, and the precipitate is now pure cadmium sulphide, possibly contaminated with bismuth sulphide.

Weighing as Cadmium Sulphate.—The cadmium sulphide is filtered off and dissolved as before. In this stage the liquid is caught in a small beaker and, after addition of 5 c.c. of dilute sulphuric acid, it is evaporated on the hot plate until fumes appear. After cooling and dilution, it is transferred to a weighed platinum basin of about 100 c.c. capacity, evaporated to dryness on the hot plate, and ignited in a low temperature muffle for a few minutes, to drive off all traces of sulphuric acid. The increase in weight of the dish gives cadmium sulphate and bismuth sulphate, together with residue from the acids used in this stage. It is therefore necessary to know the volume of acid used and to carry out a blank test, pouring the same volume of acid through a pulp filter made from the same pulp, evaporating and igniting as described. The blank may amount to 0·02 per cent. on 5 grms. taken, and therefore cannot be ignored. The net weight of cadmium sulphate includes the bismuth which has escaped deposition on the cathode in the electrolysis. Bismuth follows cadmium throughout after that stage. No brasses have been met with containing so much as 0·01 per cent. or even 0·005 per cent. of bismuth. It is, therefore, not necessary to consider the separation of bismuth and cadmium. Since some of the bismuth is deposited on the copper, it is hardly likely that the cadmium sulphate would ever be appreciably contaminated. Bismuth can easily be estimated in the residue, after weighing, by the colorimetric method with potassium iodide.

CONCLUSION.—The process is lengthy and the manipulation considerable, but the purity of the cadmium sulphide finally obtained is assured and the loss by manipulation is negligible.

DISCUSSION.

Mr. J. MYERS, who read the paper on behalf of Mr. Etheridge, said that the chief point about the author's method was that it was designed to separate very small quantities of cadmium when very large quantities of other metals were present. Copper and lead having been removed, the problem resolved itself into separating cadmium sulphide from large quantities of zinc. The method meant a lengthy manipulation, several hydrogen sulphide separations being involved, and, in his opinion, it compared unfavourably with the method published by Barr in the *J. Soc. Chem. Ind.* (1924, **43**, 24), which had been evolved for the estimation of small quantities of cadmium in zinc ores. From the results of his experience that method was twice as quick as the author's method, and he suggested that Mr. Etheridge should try Barr's method.

Dr. B. S. EVANS said that, so far as he knew, Mr. Etheridge had not seen Barr's paper, and he would certainly draw his attention to it.

The length of the process was undoubtedly a serious drawback, but Mr. Etheridge had told him that, in his experience, the Admiralty bronzes did not contain cadmium, and this, in practice, cut down the number of hydrogen sulphide precipitations. He hoped to publish shortly a method for elimination of tin that would in any case render unnecessary the three alkaline sulphide separations, but all methods of cadmium estimation had the common drawback of the uncertainty of the last weighing; it was necessary to keep the temperature of ignition between that at which sulphur trioxide was volatilised and that at which cadmium sulphate was decomposed, and these limits seemed to be rather narrow. The high blank obtained with the acids was another source of trouble.

Dr. W. R. SCHOELLER (in a written communication) said that the process described was tedious, but that the estimation of cadmium in brass was bound to be troublesome, especially if tin were also present. The only criticism he had to offer was on the statement that, if copper were precipitated as thiocyanate, it was almost certain that a large proportion of the cadmium would be occluded in the precipitate. The precipitation of copper as thiocyanate was a very valuable reaction, extensively applied in metallurgical laboratories; the separation, by this means, of copper from many other metals had been investigated by Hampe and others, and found to be satisfactory. Therefore, a statement calculated to cast doubt on the accuracy of a well-known analytical method should not be made unless substantiated by experimental evidence.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

INVERT SUGAR AS A REAGENT FOR BORIC ACID ESTIMATIONS.

THE writer was the first to show the effectiveness and cheapness of invert sugar as a reagent in the titration of boric acid (ANALYST, 1921, **46**, 3). Since then Böeseken and Couvert (*Rec. trav. chim.*, 1921, **40**, 354) and, more recently, Mellon and Morris (*J. Ind. Eng. Chem.*, 1924, **16**, 123) have arrived at similar conclusions. There should now be no hesitation in adopting invert sugar instead of the older and more expensive reagents—glycerin and mannitol.

It has been found more convenient to prepare the reagent by inverting with sulphuric acid rather than with hydrochloric acid, because in routine analysis, particularly in the case of butter and margarine, it is often necessary to estimate salt as well as boric acid in the same solution, the boric acid being estimated first; and, if the inversion has been done with hydrochloric acid, the invert sugar will contain a small amount of chloride, an allowance for which would have to be made.

The following method for making a laboratory stock solution will be found useful:—Dissolve 7lb. of commercial granulated sugar in 1 litre of distilled water and boil the solution for a few minutes until clear (it is best to do the operation in a large tin). Remove the source of heat and add quickly 25 c.c. of 3 *N* sulphuric acid from a beaker, stir for half a minute, and then add 1½ litres of distilled water in which has been previously mixed 25 c.c. of 3 *N* sodium hydroxide solution, again stir and cool. The solution should be neutral and almost colourless.

The resulting volume is about 4½ litres and the solution contains approximately 55 per cent. of invert sugar. Three c.c. of this solution are sufficient to enable 10 c.c. of 0·1 *N* boric acid solution to be titrated.

G. VAN B. GILMOUR.

THE COMPOSITION OF COCONUT OIL.

NOTWITHSTANDING the apparently good results which I have obtained by the fractional distillation of the methyl esters of coconut oil, I find myself more or less in agreement with the conclusions of Professor Drummond (ANALYST, 1924, **49**, 311) and of Mr. Elsdon (ANALYST, 1924, 423).

The assumption made by certain American investigators and others that the individual fractions obtained in the distillation consist solely of two esters, together with oleic ester, is without any scientific justification. In my work, duplicate distillations yielded results which agree very closely, but this I am inclined to attribute to the fact that the two operations were carried out under identical conditions (*i.e.* as regards the apparatus used, rate of distillation, etc.) rather than to the accuracy of the method. Hence duplicate fractionations made by the same observer will probably show a much closer agreement than results obtained in different laboratories. I am also of opinion, in agreement with Mr. Elsdon, that the differences in the composition of different samples of coconut oil, as judged by their analytical characteristics, are not sufficient to account for the variations in the quantitative results.

Mr. Elsdon quotes in his paper (*loc. cit.*) a quantitative analysis of coconut oil, referred to by Dr. E. F. Armstrong and Mr. John Allan in the recent Presidential address to the Society of Chemical Industry (*J. Soc. Chem. Ind.*, 1924, **43**, 207T), and he evidently believes that these figures have been obtained by the method of alcoholysis. This is not the case, for the figures are those published by Paulmeyer (*La Savonnerie Marseillaise*, 1907, 78), and were obtained by fractional distillation of the fatty acids in steam.

This may have caused Mr. Elsdon to be more severe in his condemnation of the method of alcoholysis than he would otherwise have been, for it cannot be gainsaid that many workers of repute seem to have obtained very fair results with the method; besides which, the results of my own work are not so widely different from those of Elsdon, though they differ more from those of Paulmeyer.

While admitting, therefore, the unsound theoretical aspect of the method, I am inclined to the opinion that the method gives approximate values when applied to coconut oil or palm-kernel oil, and that the degree of accuracy is within

±10 per cent. of each fraction, *i.e.* the error on a figure of 50 per cent. would be of the order of ±5 per cent., for a 10 per cent. component, ±1 per cent., etc.

As an example of the figures obtained with samples of different origin, the following results given by deodorised coconut oil refined in Hull and in Holland, respectively, may be quoted:

The oils had the subjoined analytical characteristics:

	Sapon. value.	Iodine value.	Reichert-Meissl value.	Polenske value.	Kirschner value.	M.Pt. °C	Free fatty acid (as lauric) Per cent.
A. English	258·0	9·2	7·15	16·0	1·81	25·3	0·05
B. Dutch	258·9	8·0	7·54	16·6	1·59	26·4	0·08

The methyl esters, prepared by the method of Haller, were freed from unchanged alcohol and distilled from a 1 litre Claisen flask, and the final redistillation was carried out in a 250 c.c. Ladenburg flask. The results of the final distillation were as follows:

A.

Fraction.		Temperature. °C.	Pressure.	Weight. Grms.	Sapon. value.	Iodine value.
Added A+B	1	up to 172	atmos.	4·79	366·0	—
„ C	2	172–209	„	34·08	335·1	—
„ D	3	130	12–14 mm.	34·58	287·0	·02
„ E+F	4	130–150	12 mm.	158·97	259·2	·40
„ G	5	150–180	„	72·69	232·1	7·55
„ H	6	180–200	„	34·41	202·1	40·25
original residue	7	200–206	„	10·58	192·1	61·09
	8	—	„	9·83	175·8	53·90
			Total	359·93		

B.

Fraction.		Temperature. °C.	Pressure.	Weight. Grms.	Sapon. value.	Iodine value.
Added A+B	1	up to 172	atmos.	1·80	381·5	—
„	2	172–200	„	23·10	346·3	—
„ C	3	99–130	14 mm.	39·44	315·0	·04
„ D	4	130–150	„	205·00	264·6	·21
„ E+F	5	150–180	„	103·40	237·2	4·57
„ G	6	180–200	„	56·66	202·3	38·75
„ H+I	7	200–218	„	9·97	192·4	54·30
Residue	8	—		7·30	211·6*	33·20
			Total	446·67		

By the use of the now discredited assumption as a basis for the calculation, conventional figures for comparison of these oils were obtained.

	Caproic acid. Per cent.	Caprylic acid. Per cent.	Capric acid. Per cent.	Lauric acid. Per cent.	Myristic acid. Per cent.	Palmitic acid. Per cent.	Stearic acid. Per cent.	Oleic acid. Per cent.	Loss Per cent.
A	0·2	7·4	9·5	49·1	17·6	4·3	1·2	10·3	0·5
B	0·2	7·2	10·7	48·7	17·5	5·4	0·8	9·0	0·8

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* This evidently contained unchanged coconut oil.

Duplicate determinations gave results agreeing closely with these.

An attempt was made to fractionate the fatty acids themselves of sample B, but, owing to difficulties caused by solidification of the distillates, the distillation was only taken up to 137° C. under a pressure of 1 mm. The approximate composition of the three first redistilled fractions was as follows:—Caproic acid, 0·2; caprylic acid, 7·3; and capric acid, 10·1 per cent.

In the main, the results obtained with these two samples of coconut oil agree with those of Elsdon (ANALYST, 1913, 39, 8).

W. N. STOKOE.

NOTE.

Claisen flask.—This vessel (employed for the preliminary distillation) is a 1-litre flask with a straight neck, into which is fused a second neck, in the form of a half-U tube, which carries the thermometer, and has the delivery outlet fused into it. The other neck of the flask is fitted with a cork holding a finely-drawn out tube, by means of which a current of air can be drawn in, while, at the same time, the pressure can be accurately regulated with a screw clip and rubber tube fitted to this inlet.

Ladenburg flask.—This vessel, of 250 c.c. capacity, consists of a bulb 8 cm. in diameter and a neck 24 cm. in length, blown out in the form of a fractionating column of 3 bulbs, each of 4 cm. diameter. The delivery tube is fused into the straight portion of the neck above the top bulb, and for this flask, thermometer and air inlet tube are fitted in the one cork.

Receiver.—This is of the type described by Lewkowitsch (*Chem. Tech. of Oils, Fats and Waxes*, 5th Ed., Vol. I., p. 664). In order to prevent loss of uncondensed distillate, a small condenser with double surface was included between the receiver outlet and the vacuum pump, and, to avoid variation of pressure when changing the distillate, it was necessary to use two vacuum pumps, one connected with the upper receiver through the condenser, and the other direct with the lower receiver.

In work of this nature extraordinary precautions must be taken to avoid loss from imperfect condensation of the lower fractions.

TOTAL SULPHUR IN RUBBER.

IN the "Standard Methods for Analysis of Rubber Articles," abstracted in the July ANALYST (p. 352) a method of ascertaining the amount of "total sulphur" is proposed. For this purpose 0·5 grm. of the rubber is treated for one hour with 15 c.c. of nitric acid saturated with bromine, then heated on the water-bath for a further hour, and evaporated to dryness. The residue is treated with 3 c.c. of nitric acid and 5 grms. of sodium carbonate, dried and fused, and the amount of sulphate is estimated by precipitation as barium sulphate. An alternative process is given which is also open to adverse criticism, but it is to the first one that most exception can be taken, for there is a risk of loss of sulphate as sulphuric acid during the earlier part of the process. For many years I have always added potassium nitrate during the treatment with nitric acid; this fixes the sulphuric acid formed, and the result is much more satisfactory. It is also desirable to eliminate the nitric radicle by evaporation once or twice with hydrochloric acid before proceeding to the final precipitation stage.

F. H. ALCOCK.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

REPORT OF THE PUBLIC ANALYST FOR THE SECOND QUARTER, 1924.

DURING the quarter 1414 samples were examined, of which 1218 (1024 informal) were examined under the Food and Drugs Acts. Of the informal samples, 50, and of the formal, 22, were adulterated.

MILK.—Thirty-eight of the 495 informal samples, and 20 of the 188 formal samples, were found to be adulterated.

COFFEE, CHICORY.—One of 26 informal samples of coffee contained about 25 per cent. of chicory. Three of the 20 samples of chicory were adulterated, one yielding 8·5 per cent. of ash, including 2 per cent. of sandy matter. One informal and one formal sample were sold as chicory, but were coffee of inferior quality (a war-time residue) which had only been slightly roasted. The vendor was fined 10s.

FLOUR.—Four of 14 samples of flour contained about 3 parts of potassium persulphate per 100,000. The matter was communicated to the Ministry of Health, who referred it to the Preservatives Committee.

CUSTARD POWDER.—Seven informal samples were, as usual, coloured and flavoured maize or sago flours. One was labelled "Extra Creamy Custard Powder," but, as it only contained 0·2 per cent. of fat, the claim was not justified.

SENNA LEAVES.—One of 15 informal samples consisted of senna pods, and the vendor was cautioned. Thirteen other samples yielded from 8·4 to 11·6 per cent. of ash. The leaves of one sample yielded 11·9 per cent. of ash, and the packet also contained 3·1 per cent. of small stones, so that the total mineral matter was 3 per cent. above the B.P. limit. The vendor was cautioned.

SOLUTION OF FERRIC CHLORIDE.—Chloride of iron is required by the B.P. to contain 5 per cent. of iron. Five of the 10 samples contained from 4·5 to 5·4 per cent., and were passed as genuine. One contained 6 per cent. and four others contained 5·7 per cent., and the vendors were cautioned. The Pharmacopœia directions for making a solution which is four times stronger are unsatisfactory. The specific gravity given as "about 1·49" does not agree with the required strength, and it seems probable that manufacturers have been misled by the directions.

SULPHUR OINTMENT.—One informal and one formal sample contained 19 per cent. of sulphur and were prepared with a paraffin basis. The vendor was prosecuted, and the magistrates dismissed the case, on payment of costs, under the Probation of Offenders' Act.

J. F. LIVERSEEGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

SALE OF SPIRITS WITH NOTICE OF DILUTION.

PRESTON *v.* GRANT.

ON November 4 an appeal from a decision of Warwickshire justices was heard in the High Court before the Lord Chief Justice and Justices Shearman and Salter.

The appellant, an inspector under the Food and Drugs Acts, had bought half a pint of whiskey from the respondent, the licensee of a publichouse, and the sample, upon analysis, had been found to be 42·26 deg. under proof.

When the sale was made there was a notice in the bar, and a similar notice in a room at the back of the bar, which read: "All spirits sold on this establishment are diluted, and no alcoholic strength is guaranteed." The appellant had not seen this notice, nor had his attention been directed to it.

The Warwickshire justices considered that the printed notice was a sufficient notification to the appellant, and that, accordingly, even though he had not seen it, he had not been prejudiced by the sale.

The Lord Chief Justice, after hearing the arguments of counsel on each side, said that the question turned upon the opinion expressed by the justices that the notice was a sufficient notice where it was found, as a fact, that the purchaser did not see the notice and had not been told of it. In other words, was a constructive notice sufficient?

In the present case, it was obvious that the sale was to the prejudice of the purchaser, unless one took the view, disregarding the Sale of Food and Drugs Acts, that the weaker the mixture, the less the purchaser was prejudiced. The seller must show that the purchaser was not prejudiced, and an inspector must not be attributed with more knowledge than an ordinary purchaser. If the article was not of the nature, substance and quality demanded he was prejudiced.

A practice had grown up of exhibiting notices intended to serve as an answer to Sec. 6 of the Sale of Food and Drugs Act, 1875, and the use to which these notices were put was that the seller claimed that the purchaser was not prejudiced because he had had notice of the difference between that which he demanded and that which he received.

So far as he was aware, there was no case which said that it was a good answer for the seller to say that he exhibited a notice which the purchaser did not see, to which his attention was not called, and of the existence of which he did not know. The case of *Pearks, Gunston & Tee, Ltd.* v. *Houghton* (1902) was a very different case from this one. In that case the purchaser got what he demanded, whereas the present appellant got something very different.

There was no such thing as a constructive notice in a case of this kind, and, in his opinion, the respondent was not protected. He did not think that the presumption that the purchaser had been prejudiced was in the smallest degree rebutted by the existence of a notice which he had not seen and to which his attention had not been called. In his opinion the appeal must be allowed, and the case remitted to the Warwickshire justices to convict.

Judgments to the same effect were given by the other Justices.

RODBOURN *v.* HUDSON.

ON November 19th the decision was given in an appeal heard in the King's Bench Division (the Lord Chief Justice and Justices Avory and Salter) from a decision of the Hampstead justices convicting the appellant of having sold, to the prejudice of the purchaser, rum diluted to 41½ deg. U.P. (*cf.* ANALYST, 1924, **49**, 229). For the defence it had been urged that the vendor was protected by a notice displayed on his premises, which notice, it was admitted, had been seen and read by the purchaser and by his agent who actually made the purchase. This notice read:—"All spirits sold in this establishment are of the same superior quality as heretofore, but, to meet the requirements of the Food and Drugs Acts, they are now sold as diluted spirits; no alcoholic strength guaranteed."

The Lord Chief Justice, in a written judgment, said that the justices had given their reasons why the sale was to the prejudice of the purchaser, and unless the Court could say that there was no evidence upon which such a decision could be based, they could not disturb the justices' decision.

Reviewing the decisions in previous cases, he pointed out that the decision in *Sandys* v. *Small* (1878; 3 Q.B.D., 449) was based upon the fact that the purchaser was fully aware of the nature of the article supplied to him, and the question of what was "due and sufficient" notice was not raised. In *Gage* v. *Elsey* (1883; 10 Q.B.D., 518) there had been a notice substantially in the same terms as in this case, and the conviction was quashed. But in that case the only point raised was whether Sec. 6 of the Sale of Food and Drugs (Amendment) Act, 1879, deprived the seller of a defence, in view of the decision in *Sandys* v. *Small.* It was assumed that knowledge had been brought home to the purchaser that the spirits were diluted, and the sufficiency of the notice was not discussed. Hence the decision in that case could not be regarded as an authority on the sufficiency of the notice.

In the case of *Morris* v. *Johnson* (1890; 57 J.P., 612) the question as to the knowledge of the purchaser was regarded as one of fact for the justices; and in *Morris* v. *Askew* (1893; 57 J.P., 724) it was held that mere notice was not sufficient, and that it was for the justices to decide whether the necessary information had, in fact, been conveyed to the purchaser.

In *Palmer* v. *Tyler* (1897; 61 J.P., 389) the justices had found that the sale was not to the prejudice of the purchaser, and it was held that, as this finding of fact was conclusive, the conviction could not be upheld. In *Dawes* v. *Wilkinson* it was held that, to obtain protection, the seller must give such notice to the purchaser as will inform him that there has been such tampering with the spirit as is expressed by the word "dilution."

In the case of *Taylor* v. *Elder* (1923) he (the Lord Chief Justice) had expressed the opinion that the notice (which was in practically the same terms as in the present case) was a notice of grave and calculated ambiguity, and to that opinion he still held. In the present case the notice was both ambiguous and misleading. "Superior quality" was a misleading term to apply to spirits which had been reduced below the statutory minimum, and there was no provision in the Sale of Foods and Drugs Acts requiring any one to sell spirits, whether of superior or inferior quality, as diluted spirits. The words "no alcoholic strength guaranteed" might well be understood to mean no alcoholic strength above the statutory minimum. It rested with the seller to prove that such notice had been given as would ensure that the sale was not to the prejudice of the purchaser. The two questions to be decided, therefore, were (1) What was the substance of the information which must be given? (2) Were the steps taken sufficient, in all the circumstances, to convey this information to the average purchaser? (If the purchaser did not see and was not made aware of the notice the question of its sufficiency did not arise (*cf. Preston* v. *Grant, supra*). With regard to (1) the purchaser must be told in substance that what he is getting is not what he asked for. As to (2) the actual sufficiency of the notice was a question for the justices. There was evidence here on which the justices could find that this notice would not convey the information.

The saying that "everyone is supposed to know the law" was too general; the maxim *ignorantia legis neminem excusat* could not be accepted as a defence against a breach of the law; but in considering this notice the justices would properly remember that the average customer knows nothing of the Food and Drugs Acts. For the Courts to "hall-mark" a notice, so that anyone by hanging it up could evade the Acts, would be unfortunate. The sufficiency of the notice was for the justices to decide in each case. The appeal must therfore be dismissed, and this judgment, if truly analysed, was, in his opinion, not in conflict with any of the authorities.

Mr. Justice Avory agreed with the judgment, subject to the reservation that he had grave doubts whether it could be reconciled with that in *Gage* v. *Elsey*, which had been acted on since 1883; also, he was not prepared to assent to what he understood to be the effect of the Scottish judgments (*Brander* v. *Kinnear*, *Kelso* v. *Soutar*, and *Williamson* v. *Soutar*; 1923, S.C.(J.), 42), *viz.* that the seller was not protected unless he specified in the notice the actual extent of dilution.

Mr. Justice Salter concurred with the judgment.

NEW NOTICES IN PUBLIC BARS.

According to *The Times* (Nov. 22, 1924) the licensed victuallers' associations have taken legal advice on the position created by the judgment of the High Court in the case of *Rodbourn* v. *Hudson*, and are of opinion that an amended form of notice will meet the requirements. They point out that, for many years past, it has been the custom to sell spirits for immediate consumption, diluted with water so as to be weaker than 35 degrees U.P., and a printed notice has been exhibited to the effect that the spirits sold are diluted and that no alcoholic strength is guaranteed.

In view of the decision in the case mentioned above, the printed notice affords no defence if the customer has not seen, or had his attention directed to the notice; and, even then, it is open to the magistrates to find that the notice would not convey adequate information to an average purchaser, and the High Court would not disturb a conviction based on this finding. It is not practicable, say the victuallers' associations, to sell all spirits not weaker than the statutory limit and to let the customer do his own dilution, since every customer cannot afford, and does not expect, to receive the full strength of spirit, the degree of dilution before sale being indicated more or less by the price charged. The situation will be met by changing the form of notice, and taking further steps to draw the attention of the customer to its existence, so that in no case can he plead ignorance of the fact that diluted spirits are being sold.

The notice in the case of *Rodbourn* v. *Hudson* is an obsolete one, and in later forms of notice there is no reference to the "superior quality of the spirits." Several summonses in different parts of London are pending, but all refer to the later form of notice, which reads:—"Notice to purchasers of spirits.—All spirits sold in this establishment are sold as diluted spirits, no alcoholic strength being guaranteed." It is proposed to fight these cases as being in a different category from that decided in the High Court.

PAREGORIC WITHOUT OPIUM.

On October 6, a herbalist was summoned at Blackburn for applying a false trade description to a certain mixture, contrary to Sec. 2 of the Merchandise Marks Act, 1887, and for selling a mixture to which a false trade description had been applied.

Evidence was given that the defendant had been asked for half an ounce of paregoric, and had supplied a mixture contained in a small bottle which was labelled in writing "paregoric," without qualification of any sort. Prior to the analysis it was thought that the defendant was selling a poison, and was therefore offending against the Pharmacy Acts, but, when written to upon the subject, he replied that the mixture sold by him was "paregoric" which was "free from opium," and that he had put this upon the label.

Mr. F. Wokes, B.Sc., for the prosecution, said that he had analysed the sample, and had found that it did not comply with the accepted standard of the British Pharmacopoeia, according to which paregoric must contain a considerable proportion of tincture of opium. In cross-examination, he agreed that the other ingredients of paregoric were present, and had an antiseptic property, but said that these alone would not constitute paregoric. He did not agree that the mixture could be sold as "paregoric essence"; in his opinion the essence of paregoric was the opium tincture.

Mr. Linsey, for the defence, submitted that the offence, if any, was a highly technical one. He produced the catalogue of a wholesale firm to show that paregoric was sold both with and without the addition of opium. "Paregoric essence" had been used as a favourite remedy in Lancashire for generations, and it was usually procured from herbalists. Had the defendant sold a mixture containing opium, he would have been liable to a heavy penalty, but there was a notice in his shop to the effect that no dangerous or poisonous drugs were sold there. The Merchandise Marks Act was not intended to apply to a case like this, where there was no intention to deceive or to make a profit out of the deception. The bottle and the preparation had been sold for 3½d. with the dangerous element removed.

The Bench held that while the defendant was perfectly innocent of any intention to break the law, there had been an offence. Fines of 40s. for the first case, and 20s. for the second, were imposed, and defendant was required to pay the costs of the witnesses.

Federated Malay States.

ANNUAL REPORT OF THE CHEMICAL LABORATORIES FOR 1923.

THE total number of samples examined during the year was 6521, as compared with 2702 in 1922, the increase being mainly due to samples of chandu dross and counterfeit coins.

MEDICAL DEPARTMENT.—Of the 774 samples of milk examined, 107 failed to comply with the standards prescribed in the Sale of Foods and Drugs Enactment, 1913. Eighteen of these were deficient in milk fat, 86 were deficient in non-fatty solids, and 3 in both.

WATER.—Chemical analysis of 343 samples and bacteriological examinations of 40 samples were made. The water from the Ayer Kuning reservoir has been treated with chlorine since September, 1923. The addition of 0·5 part of chlorine per million reduced the number of bacteria by 71 per cent., and 0·75 part per million by 84 per cent.

TOXICOLOGICAL ANALYSES.—Three cases of human poisoning were investigated. In one of these spirits of salt had been accidentally swallowed, and free acid and chlorides were detected in the vomit and in the stomach.

In a case of arsenical poisoning $\frac{1}{80}$ grain of arsenic was recovered from the stomach and contents, kidney and a portion of the ileum.

In the third case $\frac{1}{50}$ grain of strychnine was found in the stomach and $\frac{1}{30}$ grain in the stomach contents.

Vitamin B Extract.—The preparation of this extract from rice polishings was continued throughout the year, and 5159 fluid ounces were issued to medical practitioners, dispensaries and hospitals for the treatment of beri-beri.

TRADE AND CUSTOMS DEPARTMENT.—The total number of samples examined was 3619, as compared with 719 in 1922. These included samples of liquors for alcoholic strength determinations, toddy, chandu, chandu dross, and deleterious drugs (2 of which contained morphine hydrochloride).

POLICE DEPARTMENT.—Of 993 samples of coins and coining materials examined, 967 were counterfeit coins.

Tests for blood stains were applied in 106 cases, of which 43 gave positive results. Of these, 36 were tested for human blood by the precipitin test, and 23 gave positive reactions.

Poisons were identified in only four cases, *viz.* potassium cyanide in two cases of suicide, and arsenic in two cases.

H. MARSDEN, *Acting Chemist.*

The Cellular Content of Milk.

INVESTIGATION MADE ON BEHALF OF THE MEDICAL RESEARCH COUNCIL.*

THERE exists the greatest divergence of opinion as to the significance of the cellular elements and micro-organisms that occur in milk. Few will deny that milk should not contain pus, but what constitutes pus in milk? All milk contains leucocytes, but do these leucocytes become converted into pus cells, and, if so, how can one be distinguished from the other? The presence of a certain number of leucocytes in milk is strictly physiological, though beyond a certain point it may, probably does, become a pathological feature. One school holds firmly to the idea that the cellular contents of milk are all derived from the blood; that they are true blood cells, and that under certain conditions they constitute pus. Another school asserts that the cells of milk are tissue cells of purely epithelial origin, which under no circumstances can be spoken of as pus cells. The truth appears to lie somewhere between these extremes.

METHODS OF EXAMINATION USED.—To distinguish the various cells that may be present in milk, a differential method of staining must be used, and the contradictory statements published must be attributed to failure to appreciate this condition. The following method of cell examination has been adopted in this enquiry, and a stain used which has been found to give constant and reliable results for a differential count:—

The animal is milked in the ordinary way and at the usual times. During the milking three samples of the milk from each quarter—12 samples in all—are milked direct into sterilised test-tubes, which are plugged immediately. Of the three samples, one is taken at the commencement of milking—called the "fore-milk"—another in the middle of the operation—the "middle-milk"—and a third at the end of milking (but without any undue straining)—the "strippings." Taking each of these samples in turn, the tube is well shaken and 10 c.c. poured into a centrifuge tube of Houston's pattern the lower part of which is drawn out and accurately marked with a scale of 1/100 cm., and centrifuged at 3000 revolutions per minute for ten minutes. The cream and supernatant fluid are carefully removed, 0·5 c.c. of milk being allowed to remain above the deposit. These are thoroughly mixed with a capillary pipette on which is a bulb and into which the mixture is finally drawn up. The pipette is then sealed off and centrifuged until the deposit is carried to the bottom of the pipette, whence, with a still finer tube, it may be removed and spread on a glass slide. The film is then air-dried, stained at once by Jenner's method, examined with a Zeiss 1/12, and the cells noted, described, drawn, and counted.

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* By P. C. Varrier Jones, M.A., M.R.C.S. Abridged from *The Lancet*, 1924, 207 (Sept. 13), 537–542, by permission of the Editor of that journal and of Dr. Varrier Jones. The original report has 6 coloured diagrams of the different types of cells.

This method is not put forward as giving correct numerical results, but rather as a differential method in which the well-stained cells of different types may be readily distinguished.

The question now arises: are all the cells thrown down as sediment in the centrifuge, or are some carried upwards and held in position by the cream which rises to the surface? In normal milk it has been found that the number of cells entangled in the cream is a negligible quantity, and it is necessary in any differential count to examine only the deposit found at the bottom of the pipette.

CHARACTERISTICS OF CELLS IN MILK.—Observations have led to the conclusion that the following cells are to be found in milk obtained from a normal animal:—

1. *The Finely Granular Eosinophile Cell.*—A polymorphonuclear cell (10 to 12 μ in diameter), more or less round or elliptical, or even irregular in outline, the shape depending on various factors, such as speed of centrifuging. Clear cytoplasm remaining unstained by Jenner's method. Nucleus, as a rule, stained deep blue (old cells stain badly); branched or lobulated in form. In cells of fresh milk pseudopodia stretch out in all directions; phagocytic—the bacteria being contained in vacuoles lying in the substance of the cell. In a normal milk the number is almost negligible; but in milk from an inflamed quarter the number may be very considerable.

2. *The Coarsely Granular Eosinophile Cell.*—The coarsely granular eosinophile cell is slightly larger than the finely granular polymorphonuclear cell; it is oval or round in shape, and measures 10 to 13 μ in diameter. The cytoplasm is perfectly clear and transparent, but in this case, again, is closely studded with highly refractile granules, measuring in some cases as much as 0·5 μ in diameter; by Jenner's method these granules stain a deep pink.

The nucleus, usually of a light blue tint, is placed either in the centre of the cell or, more commonly, towards the periphery. It is either kidney-shaped or lobed, the different lobes being connected by bands of nuclear substance. In many films the cells appear to have burst; the granules are then scattered in all directions.

3. *The Large Mononuclear Leucocyte.*—The large mononuclear leucocyte is one of the largest cells met with in the deposit from cow's milk. It is round or oval in shape, invariably staining a dark blue. The stain, however, is not evenly distributed, but appears as a tangled thread throughout the substance of the cell. This is the nucleus, which fills up nearly the whole of the cell. Usually, however, there is a small portion of cytoplasm on one side of the nucleus, but it rarely makes a complete circle round it. This portion of the cell is stained less darkly, but it is nevertheless of a distinct blue colour. These cells are invariably present in all samples of cow's and goat's milk, and in some are a very prominent feature.

4. *The Lymphocyte.*—The lymphocyte is a small blue cell which, as a rule, stains deeply. The cytoplasm is small in amount and forms a thin ring of light blue colour around the darkly-stained nucleus. These cells vary in size from 2 to 4 μ, the majority measuring about 3 μ. The nucleus is round or slightly oval in shape, stains darkly, and is not reticular. It occupies almost the whole of the cell. Such cells are fairly numerous in normal milk, but appear to be found in greater numbers in certain breeds of cattle than in others.

5. *The Basophile Cell.*—The basophile cell (8–10 μ), a rare cell, is almost circular in shape, but may be oval. The cytoplasm is clear and transparent, and has scattered unevenly throughout its substance dark blue granules, large and small. The large nucleus occupies two-thirds of the cell, it is oval or rounded, and is always single. By Jenner's method it stains a pale blue. As a rule, the basophile cell is absent from films of normal milk, but under certain conditions it appears, though never in great numbers.

6. *The Large Neutrophile Cell* (5–9 μ).—The large neutrophile cell is extremely rarely met with. It is oval in shape, rarely round. The cytoplasm is stained a faint light blue throughout, the whole being filled, except in the region of the nucleus, with very fine granules, which take on a pale mauve tinge. By Jenner's stain the nucleus is tinged light blue.

7. *Red Blood Corpuscles*.—Red blood corpuscles are invariably present in a sample of cow's milk. In normal milk they are exceedingly few in number; as a rule, only one or two are found in a film made from the deposit of 10 c.c. of milk. It must be remembered, however, that in milk films the individual red blood corpuscles are not exactly similar to those found in normal blood, but they are always easily recognisable.

8. *Large Epithelial Cells*.—Large epithelial cells are almost always present. They vary greatly in size, some measuring as much as 20 μ in diameter. The cytoplasm is somewhat granular, having a ground-glass appearance; it stains a more or less deep blue with Jenner's stain. The cells are usually rounded, but may be oval, and appear to have a well-developed envelope, which takes on a slightly deeper stain. The nucleus is large, oval or rounded, occupying a quarter of the cell, and stains a less deep-blue than the rest of the cell. It usually has a mottled appearance as though composed of a chromatin network. In the cytoplasm of the cell are usually one, two, or even more clear spaces with well-defined outlines, usually round, more rarely oval in shape. These are fat globules surrounded by an envelope which prevents the fat from taking the ordinary fat stains.

9. *Mononuclear Eosinophile Cell*.—Other vacuolated cells appear from time to time in milk. Apart from the finely granulated, eosinophile cell which may become vacuolated through age, or otherwise, there are other cells which closely resemble them. The cytoplasm of these cells, instead of being clear and transparent, stains a light pink, the substance being more or less vacuolated. The nucleus stains a light blue and is usually pushed to the side of the cell by the large number of vacuoles in the cytoplasm. These cells are abundant, especially when the cow dries off; but they may be present at other times. They may, however, be unvacuolated, when they may measure 9–10 μ in diameter, but they are often less than this. The author regards this as an epithelial cell, probably derived from the secretory layer of the gland tissue.

CELLULAR CONTENT OF NORMAL MILK.—Should the cow happen to be a normal animal—*e.g.* a heifer some days after her first calf, without any complications or udder trouble after calving—the blue cells, both large and small, which occupy the field are numerous, and at once arrest attention; indeed, one is apt to make up one's mind that the milk is full of lymphocytes both of the large and small variety. Looking over the fields, one may observe here and there, not usually more than one per field, a finely granular eosinophile cell, with nucleus sharply stained, and with the granules distinctly visible; characters that enable one to pick it out without difficulty from the surrounding cells. It would be a great mistake, and very misleading, to class these cells together as pus cells; most of them are broken-down polymorphonuclear cells. Now and again a coarsely granular eosinophile cell which stains well, resembling very closely the coarsely granular eosinophile cell of ox's blood, may be seen. Again, a much larger cell, round or oval in shape, with large pale blue nucleus, the cytoplasm of the cell staining a mottled blue, may be present. Evidently these different cells cannot be lumped together as pus. Moreover, one finds in the field a number of small dots, stained deep blue, 1 μ or so in diameter, sometimes with only a tag of pink substance around the blue point, or near its edge. These are obviously nuclei extruded, possibly, from blood cells, but more probably from secretory epithelial cells of the acini. The pink portion is part of the cytoplasm of the cell which was torn away when the nucleus was extruded. From this picture, which is invariably presented in the sediment from normal milk, it is obvious that a simple count can give no information as to the condition of the secreted fluid or of the secretory tissue. The number of cells (lymphocytes), added to the stray polymorphs, coarsely granular eosinophiles and tissue cells, along with the extruded nuclei, may give a high "leucocyte" count; but under these circumstances we cannot justly claim that the cow is suffering from a diseased condition of the udder or that the fluid is "pus." A differential count of cellular deposit must always be made before a diagnosis is justified.

CELLULAR CONTENT OF ABNORMAL MILK.—A high cell count is not of necessity evidence of a diseased udder, nor, on the other hand, does a low count necessarily indicate an udder free from disease. The actual count may, of course, in certain doubtful cases help in the formation of a diagnosis, but it cannot by itself be depended upon. For the present attention is directed to the vital importance in all cytological examinations of milk, of applying this differential staining and counting method *quarter by quarter*; for it is not sufficient to make an examination of the mixed milk by this method.

INFLUENCE OF PHYSIOLOGICAL CONDITIONS.—*Breed of Cow.*—It appears that normal cows of different breeds have different proportions of cells in their milk. In the Hereford heifer the cells are of large mononuclear lymphocyte type. Large mononuclear leucocytes also appear to be present invariably in the milk from Hereford cows, but are not so noticeable a feature in the films prepared from the milk of the cows of the other breeds. The Welsh heifer's milk always contains many—indeed, they were almost the most noticeable feature—of the very small blue cells, which are almost entirely absent from the Hereford cow's milk. Owing to the large number of these small cells, the cellular content of the Welsh heifer's milk is always high. In the milk of the Shorthorn, the very small cells, so familiar by their presence in the milk of the Welsh heifer, are here seldom met with; their place is taken by cells with a pale-blue nucleus and a surrounding layer of granular protoplasm, the granules being very fine and staining blue. This divergence of cellular content still requires explanation.

Influence of Calving.—Besides the colostrum corpuscles—*i.e.* cells laden with fat globules probably derived from the epithelium of the acinus, the polymorphonuclear cells are very numerous, some staining sharply, others vacuolated and evidently in process of disintegration. These colostrum corpuscles appear in such great numbers that the stained film appears quite pink, in this respect differing markedly from the film of the ordinary milk, which has a blue tinge. Lymphocytes, both of the large and small variety, though present, are not numerous; the cell with the pale-blue nucleus and granular protoplasm (with fine blue granules) predominates. The polymorphs, even in colostrum, are actively phagocytic. As the milk becomes normal the colostrum corpuscles and the polymorphonuclear cells gradually disappear; and, if no inflammatory process supervenes, the milk cells soon return to their normal count. If, however, by any chance one quarter of the udder becomes inflamed, the polymorphonuclear variety of cell persists, and is present even when the milk from the other quarters affords no trace of its presence.

Effect of Stage of Milking.—As a rule, unless a method of milking such as the use of a milk tube is employed, the cells of all three samples are much the same as regards both type and proportion, although the absolute number may vary considerably.

Effect of Intervals in Milking.—When the interval between the morning and the evening milkings is unduly prolonged the cells increase in number, especially the polymorphonuclear cells and the red blood corpuscles.

Relationship between Fat Content and Cellular Content.—In no case during this investigation has there been found any increase in the fat content of milk where there has been a rise in the polymorphonuclear cells. The statement of Pennington and Roberts (*J. Infect. Dis.*, 1908, **5**, 72) that "in almost 75 per cent. of the herd, perfectly healthy, clean cows show *pus* at the close of the milking period, and *coincident chemical analysis*, that the percentage of butter fat is from 0·5 to 1·5 per cent. above the normal for that particular individual" has not been borne out by the present results.

Physiological "Drying-Off."—In the normal animal when drying-off occurs the increase in the number of cells comes on quite gradually; the milk yield slowly falls off, and with this there is a corresponding increase in the number of cells, especially of one definite type. The films all present common features, and with a little practice it is possible to make a diagnosis of drying-off with great confidence. Should no differentiation be made between the various cells the condition would be diagnosed as due to previous disease, probably chronic inflammation of long standing. These drying-off cells, however, are usually circular in outline; the cytoplasm, which is often "vacuolated," contains no granules, and takes on an irregular somewhat deep pink stain. The nucleus may be single, or may be bi- or even tri-lobed, with the lobes apparently not joined together. The one, two, or even more "vacuoles" present in a single cell are stained by Nile blue, and therefore contain fat.

Pathological "Drying-Off."—Any severe inflammatory trouble may result in drying-off of the quarter or quarters affected. The cells are then of several varieties, the polymorphonuclear form being found in equal or greater extent than those described above as usually present. The inflammatory process introduces an extra factor, and may alter the cellular content to such an extent that the deposit may now be described as pus:—

Phagocytosis.—An experiment is described in detail by which it was demonstrated that the polymorphonuclear cells described above were actively amœboid and phagocytic.

SOURCE OF THE CELLS.—It is held very generally that all the cells found in milk are purely blood cells, but Winkler (*Zeitsch. Landw. Vers., Oesterreich*, **1908**, **9**, 562) and his school maintain that they are entirely of epithelial origin. On the one hand, we have such statements as the following: "The outstanding feature in the examination of some hundred films prepared both from normal cows at different periods of lactation and from cows presenting slight signs of mastitis and representing many thousands of cells, is that *no cell having any decided resemblance to a polymorphonuclear leucocyte has been detected*, and phagocytosis of bacteria present is conspicuous by its absence." (Hewlett, Villar and Revis, *J. Hygiene*, **1909**, **9**, 271; **1910**, **10**, 56; **1911–12**, **11**, 97; **1914**, **13**, 87.) It is obviously difficult to concur in this view, the polymorphonuclear cells described in this paper being identical with those found in the blood.

These leucocytes must be regarded as pus cells when in the course of time they break down and disintegrate. The slightest inflammation of the udder is followed by the appearance of a considerable number of these cells the characters of which are always the same, and the phagocytic activity of which, when examined fresh, can be demonstrated. Many of the author's drawings show this very clearly, and he cannot agree with those observers who say that phagocytosis "is conspicuous by its absence." He is, however, in agreement with those who point out that the cell with a single oval or rounded nucleus, which stains well but not always uniformly, the cytoplasm of which is structureless and which stains well with eosine, is derived from the secretory layer of the gland tissue. This cell, which he regards as epithelial in type, is present in large numbers when the gland tissue ceases to perform its proper function as the cow "dries off." On account of its sometimes having a double nucleus it is apt to be mistaken for a polymorphonuclear cell, and by many authors it is regarded as a pus cell. The small blue cells described above as lymphocytes are really the lymphocytes of the blood-stream. Again, the coarsely granular eosinophile cells and the basophile cells which are so often present in milk undoubtedly correspond to similar cells found in the blood.

The other cells are epithelial in type, and a few are undoubtedly derived from lymphoid tissues.

Presence of the Red Blood Corpuscle in Milk.—It would not be accurate to say that *every* sample of milk contains red blood cells, but it is undoubtedly true that 99 per cent. of the milk samples which the author has examined have contained erythrocytes. Even in normal milk—using the term in its strictest sense—he has met with an occasional red blood corpuscle. In acute congestion of the udder red corpuscles make their appearance, and they are always present also in the first milk or colostrum after calving.

Causes of the Presence of Blood in Milk.—The commonest cause of the presence of blood in milk is said to be rupture of a blood-vessel. This may be true where large numbers of erythrocytes are met with, but it is certainly not the case where the blood is detected only on microscopic examination of the milk. Apart from pathological and mechanical sources of blood, it is not generally recognised with what ease the red blood corpuscle can get into the milk-stream, and the fact that this can occur is undoubtedly a factor to be taken into consideration when one considers the possibility of pathogenic organisms—*e.g* the tubercle bacilli getting into the milk-stream by the same route.

So far as these observations go, it is evident that the cells in milk are partly blood cells, partly lymphoid, and partly epithelial. While it is impossible to agree with the statement that "no cell having any decided resemblance to a polymorphonuclear leucocyte" is present in milk, it is obvious that to concede that all the cells in milk are pus cells is equally out of the question. Although it must be admitted that at certain times, and under certain conditions, pus cells are found in milk, it does not by any means follow that, whenever the cell count is high, we are dealing with a purulent condition.

In this report an endeavour has been made to point out that, although reliance cannot be placed upon a quantitative leucocyte standard, yet, by means of the differential count, it is possible to distinguish all the various conditions most accurately, and in many cases even to make a diagnosis, and to set up a qualitative standard which may be of great service. It is, indeed, necessary not only to modify the existing standards, but to revise our whole method of diagnosis. The method here described, together with the results of the investigation, indicates a method of procedure which certainly promises to give more satisfactory results as regards diagnosis than have yet been obtained.

United States Department of Agriculture.

FOOD INSPECTION DECISIONS 193, 194, 195.

THE following revised and amended definitions and standards for cider vinegar, marjoram and cumin seed were adopted by the Joint Committee on Definitions and Standards, composed of representatives of the United States Department of Agriculture, the Association of American Dairy, Food and Drug Officials, and the Association of Official Agricultural Chemists, August 10, 1923, and Feb. 27, 1924.

(193). Vinegar, cider vinegar, apple vinegar, is the product made by the alcoholic and subsequent acetous fermentations of the juice of apples, and contains in one hundred cubic centimetres (100 c.c.) (20° C.) not less than four (4) grms. of acetic acid.

(194). Marjoram, leaf marjoram, is the dried leaves, with or without a small proportion of the flowering tops, of the *Majorana hortensis* Moench. It contains not more than sixteen per cent. (16%) of total ash, not more than four and five-tenths per cent. (4·5%) of ash insoluble in hydrochloric acid, nor more than ten per cent. (10%) of stems and harmless foreign material.

(195). Cumin seed is the dried fruit of *Cuminum cyminum* L. It contains not more than nine and five-tenths per cent. (9·5%) of total ash, not more than one and five-tenths per cent. (1·5%) of ash insoluble in hydrochloric acid, nor more than five per cent. (5%) of harmless foreign matter.

The foregoing definitions and standards are adopted as a guide for the officials of this Department in enforcing the Food and Drugs Act.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Changes in the Optical Activity of Sucrose produced by Heating. M. A. Rakusin and A. N. Nesmejanow. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 151–152.)—Caramelised sugar has become increasingly important as a constituent of diabetic foods. An optically active, Fehling-reducing product, of coffee-brown colour and sweet taste, is best prepared by heating cane-sugar to 150° to 160° C. for 2 to 4 hours. The effect of heat on a sugar having m.pt. 178–183° C. and $[\alpha]_D = +65{\cdot}33$ was as under:

Temperature.	Time.	$[\alpha]_D$.	Colour.
m.pt.	—	+48·33	pale yellow
m.pt.	—	+59·33	" "
190°	½ hour	—	black
180°	"	0	"
160°	"	+53·66	pale yellow
170°	"	+22·66	dark brown
160°	1 hour	+15·00	pale brown
150°	2 hours	+13·20	" "
140°	4 hours	+21·00	yellow

H. E. C.

Modifications of the Picric Acid Method for the Estimation of Sugars. J. J. Willaman and F. R. Davison. (*J. Agric. Res.*, 1924, **28**, 479–488.)—There is no reasonably permanent colour standard suitable for use in the picric acid method of sugar estimation. The best available is a 0·08 per cent. glucose or 0·076 per cent. sucrose solution in saturated picric acid; these will keep for about a week. Careful examination of the method brings out the fact that strict attention to empirical detail is essential for accuracy, and the following procedure is recommended. Solutions required are:—Saturated recrystallised picric acid, 20 per cent. (anhydrous) sodium carbonate and the above-mentioned standard sugar solutions. The solution to be examined is first clarified with lead acetate, de-leaded with sodium phosphate, and diluted so that the sugar content is between

0·02 and 0·24 per cent. Then, for the estimation of reducing sugars, 1 c.c. of the solution, 2 c.c. of saturated picric acid solution and 1 c.c. of the sodium carbonate solution are mixed in a test tube closed with a tin-foil covered cork and heated in boiling water for 20 to 30 minutes, cooled, and diluted to 10 c.c. For the total sugars, 1 c.c. of the solution and 2 c.c. of the picric acid are heated in the same way for 10 minutes, then 1 c.c. of sodium carbonate solution is added, and the heating continued for 20 to 30 minutes and, as before, diluted to 10 c.c. The colour so obtained is matched in a colorimeter with that of 2 c.c. of the standard sugar solution similarly treated. The amount of sugar is not quite proportional to the intensity of colour, though it is nearly so. The colour values, as compared with glucose = 1·000, of the various sugars, and the appropriate factors are:

Sugar.	Colour value.	Conversion factor.	Sugar.	Colour value.	Conversion factor.
Glucose	1·000	1·000	Rhamnose	1·298	0·774
Fructose	1·000	1·000	Lactose	0·750	1·333
Maltose	0·770	1·298	Arabinose	1·000	1·000
Mannose	1·000	1·000	Xylose	1·071	0·934
Galactose	0·883	1·132			

A special table is given showing the exact colorimeter readings corresponding to varying amounts of sugar. H. E. C.

Limits for the Detection of Traces of Alkali or Soap in Refined Coconut Oil. W. L. Brooke. (*Philippine J. Sci.*, 1924, **25**, 151–153.)—Alkali was introduced in varying proportions into refined coconut oil previously washed three times with distilled water. The oil was then ashed, the ash dissolved in distilled water, and a drop of phenolphthalein solution added. A pink coloration was taken to indicate the presence of alkali or soap in the oil. Less than 0·02 per cent. of sodium carbonate was thus detected, and 0·00065 per cent. of sodium hydroxide with certainty, and as little as 0·00033 per cent. of the latter gave a pale pink coloration. D. G. H.

Non-Volatile Acids of the Peach. E. K. Nelson. (*J. Amer. Chem. Soc.*, 1924, **46**, 2337–2338).—According to Kunz and Adam (*Zeitsch. Nahr. Genussm.*, 1906, **12**, 670) the peach contains citric acid, but no malic acid. Bigelow and Dunbar, however (*J. Ind. Eng. Chem.*, 1917, **9**, 762), concluded that probably only malic acid is present. In experiments to settle the question 18 kilos. of sliced peaches were boiled with water and the extract concentrated. The pectin was precipitated with alcohol, and the filtrate treated with lead acetate. The lead salts were decomposed with sulphuric acid, and the liberated acids were converted into their ethyl esters, which were then fractionally distilled at a pressure of 10 mm. The acids in the fractions were identified by means of their hydrazides, 0·7 grm. of the ester being dissolved in 3·5 c.c. of absolute alcohol and treated with 0·5 c.c. of hydrazine hydrate. A hydrazide melting at 175° to 178° C. was identified as *l*-malic dihydrazide, and one melting (not sharply) at 145° C. as the trihydrazide of citric acid. The amounts of these compounds obtained indicated that the non-volatile acids of the peach consist principally of a mixture of *l*-malic acid and citric acid in almost equal proportions.

New Method of Estimating Allyl-isothiocyanate. Morvillez and Meesemaecker. (*J. Pharm. Chim.*, 1924, **30**, 236–240.)—One hundred c.c. of water are added to 5 grms. of powdered mustard previously mixed with 20 c.c. of 90 per cent. alcohol, and the whole left to stand at 35° C. for 1 hour in a corked flask. The mixture is then distilled into 20 c.c. of ammonium hydroxide solution until 100 c.c. of liquid are obtained. To 50 c.c. of the distillate, neutralised and subsequently acidified with 10 c.c. of sulphuric acid, 10 c.c. of 0·1 *N* iodine solution are added, and the whole left in the dark for 15 minutes and titrated with q c.c. 0·1 *N* sodium thiosulphate solution. Then the percentage of allyl-isothiocyanate present is $(10-q) \times 0{\cdot}00495 \times 4 \times 10$. This method is more rapid than the various silver methods and as accurate. The presence of white mustard does not interfere with the reaction.

D. G. H.

Estimation of Peroxide in Ether. A. W. Rowe and E. P. Phelps. (*J. Amer. Chem. Soc.*, 1924, **46**, 2078–2085.)—The method described depends on the reaction between a peroxide and cadmium and potassium iodides in acid solution. Ten c.c. of the ether sample are mixed with 15 c.c. of aqueous 5 per cent. cadmium iodide and potassium iodide solution, 15 c.c. of dilute sulphuric acid, and 25 c.c. of pure alcohol; after about one hour, the liberated iodine is titrated with 0·1 *N* thiosulphate solution. A control experiment is made at the same time, using the same quantities of reagents but without the ether. The alcohol used is purified by treating each litre of 95 per cent. alcohol with 4 grms. of *m*-phenylenediamine hydrochloride, allowing the mixture to stand for several days and distilling it slowly.

W. P. S.

Biochemical, Bacteriological, etc.

Chemotherapeutic Experiments with Chaulmoogra and Allied Preparations. O. Schobl. (*Philippine J. Sci.*, 1924, **25**, 123–134; 135–150.)—Water-soluble compounds; sodium, copper and nickel salts of fatty acids; and double bond containing compounds (particularly with the double bond in the side chain of the ring in aromatic compounds) were all found to be antiseptic, but the most powerful class was that of the unsaturated alcohols containing phenyl groups. The antiseptic effect of phenols increased with the number of hydroxyl groups present, specially with those in the ortho position, and also with alkylation *on the ring*. The amino group had a reverse effect on the antiseptic powers of aromatic compounds, but when linked directly to the side chain was strongly antiseptic. Open chain terpenes were found to be more antiseptic than their cyclic isomers.

No relationship was found to exist between the degree of unsaturation of vegetable oils which stimulate or inhibit the growth of acid-fast bacteria, and their growth-stimulating or inhibiting activity, but in those which stimulate the growth of acid-fast bacteria the condition of unsaturation is paramount, so that saturated chaulmoogra oil lacks the activity of the unsaturated oil, and, further, its growth-inhibiting activity is lost if the ring structure is altered by saturation with hydrogen. There are indications that acids from the chaulmoogric series with short side chains are more effective *in vitro* than those with long chains, but acid-fast bacteria will, after a time, withstand larger doses of the acids than at first. The growth-stimulating effect of certain vegetable oils was found to be due to the glyceryl and not to the acid part of the oil. D. G. H.

Colour Reactions of Antirachitic and Antiscorbutic Principles. Bezssonoff. (*Comptes rend.*, 1924, 572–574.)—Antirachitic principles may be identified by the orange colour produced on mixing a benzene solution of the fatty material with an aqueous solution of phosphomolybdotungstic acid, and the colour is intensified by prolonged heating and by oxidation by air. With antiscorbutic principles a blue coloration develops, but the reaction is not so well defined as that for antirachitic bodies. Cod-liver oils, egg yolk and butters were tested, the latter showing pale yellow colorations of the benzene layers, and blue aqueous layers. Olive and arachis oils and lard were found devoid of the fat-soluble factors, but a feeble blue coloration was apparent in the case of unheated olive oil (*cf.* ANALYST, 1921, **46**, 462). D. G. H.

Antiscorbutic Value of Fresh and Canned English Tomatoes. E. M. Delf. (*Biochem. J.*, 1924, **18**, 674–678.)—The minimal daily ration of raw fresh juice of English tomatoes protecting young guinea pigs from scurvy has been shown to lie between 1·5 and 2·5 c.c.; thus it is not quite as effective an antiscorbutic as fresh orange or lemon juice. The locally produced S. African tomato is decidedly inferior to the English fruit, the minimum protective dose under similar experimental conditions being about 4 c.c. daily. A sample of bottled tomatoes, which had been stored for six months, was found to have lost about two-thirds of its original value. By canning, by a method described, a loss of nearly three-quarters of the original value was sustained, and a further but smaller loss occurred after storage of the canned fruit for nearly 4 years. Canned tomato purée had lost about seven-eighths of the value of the original juice. The concentrated juice is, bulk for bulk, but little superior to the canned unconcentrated juice, when prepared as described in the paper. P. H. P.

Antiscorbutic Fraction of Lemon Juice. II. S. S. Zilva. (*Biochem. J.*, 1924, **18**, 632–637.)—In a previous communication Zilva (*Biochem. J.*, 1924, **18**, 182) showed that it is possible to remove the acids and sugar from lemon juice without perceptibly influencing its potency. A series of experiments is described in which some very active antiscorbutic fractions have been prepared. It is shown that still more extraneous matter may be removed without vitiating the activity of the antiscorbutic principle. The most potent preparations contain only 0·03–0·07 per cent. of solid matter in solution, while retaining approximately the entire activity of the original juice. A table gives the scheme of fractionation. The fractions were tested on guinea pigs, and small daily doses were found to protect them from scurvy. One fraction was also tested curatively on monkeys which had been kept on a scorbutic diet and had developed scurvy. The fraction cured them. All the active fractions reduce ammoniacal silver nitrate and decolorise potassium permanganate. The purest preparations showed an extremely low nitrogen and phosphorus content. P. H. P.

Differential Dialysis of the Antiscorbutic Factor. II. S. J. B. Connell and S. S. Zilva. (*Biochem. J.*, 1924, **18**, 641–646.)—Adopting the technique of Brown (*Biochem. J.*, 1915, **9**, 591), Zilva and Miura (*Biochem. J.*, 1921, **15**, 422), showed that the antiscorbutic factor of lemon juice diffuses through collodion membranes which permit the passage of substances of such molecular dimensions as methylene blue, neutral red and safranine. The vitamin diffuses also through collodion membranes of higher permeability. The authors have extended this investigation in the hope of utilising this method of differential dialysis for the purpose of separating the active principle from extraneous matter, but this has not been accomplished. Their experiments are described and results given. They find that the diffusion of the antiscorbutic factor proceeds differently from that of the sugar and nitrogenous substances present in decitrated lemon juice, thus confirming the observations of Zilva (*Biochem. J.*, 1924, **18**, 182; **18**, 632) in this connection. The antiscorbutic vitamin in swede juice and in lemon juice diffuses through membranes of the same permeability. This suggests that the factor in both sources is of the same molecular magnitude. The vitamin will diffuse through collodion membranes of somewhat lower permeability than those which permit the passage of the above-mentioned dyes. Indications suggest that the size of the active molecule cannot be far removed from that of the hexoses.
P. H. P.

Influence of Diet and Sunlight on Vitamins of Milk. E. M. Luce. (*Biochem. J.*, 1924, **18**, 716–739.)—The experiments which were carried out on a Jersey cow are described in detail, and the results are discussed. Milk from the same cow was shown to vary within wide limits as regards its antirachitic and growth-promoting power. The diet of the cow appeared to be the main factor in determining the growth-promoting value of the milk. When the cow received a diet of fresh green grass the milk possessed a higher growth-promoting value than when she received dry fodder deficient in fat-soluble vitamins. This result was obtained irrespective of whether the animal was kept out of doors in sunlight or in a dark stall. The evidence as to whether light does, or does not, contribute to the action, is not conclusive from these experiments. The antirachitic value depends on the diet of the cow, and possibly also on the degree of illumination to which she is exposed. Milk from a pasture-fed cow has a definite and high antirachitic value; the same animal, when stall fed in the dark, yielded a milk much inferior in antirachitic properties. It is conceivable that, in the absence of sunlight, it would be impossible to raise the antirachitic value of the milk above a certain level. A possible contributory cause of the seasonal incidence of rickets in children, may be the use of milks the antirachitic value of which is higher in summer than in winter. P. H. P.

Yeast Growth-Promoting Vitamin Tested for its Effects on Animals. J. Deas. (*J. Biol. Chem.*, 1924, **61**, 5–8.)—The yeast growth-promoting vitamin, first described by Wildiers (*La Cellule*, 1901, **18**, 313) as a "bios," was long considered to be the same as vitamin *B*. Funk and Dubin (*J. Biol. Chem.*, 1921, **48**, 437) showed that the yeast growth-promoting vitamin, called by them vitamin *D*, can be separated from that which causes growth of rats. Lucas (*Science*, 1924, **59**, 197) found malt rootlets to be a more abundant source of bios than malt. The purified bios has now been separated into two fractions—Bios I. and Bios II. The former is precipitated by baryta, is not adsorbed on charcoal, and is not precipitated by lead acetate except in the presence of excess of ammonia; the latter is adsorbed by charcoal, can be removed from its solution by shaking with yeast, and is soluble in acetone. Either of these two substances by itself, and especially Bios II., is detrimental to the growth of yeast. The feeding experiments with rats, described in detail, show that malt rootless contain little or no vitamin *B*. Neither Bios I. or II., nor both in a combination suitable to promote an abundant growth of yeast, will promote the growth of rats. Consequently, Wildiers' bios and Funk's vitamin *D* are not identical with the rat growth-promoting vitamin *B*. It is also shown that the "bios" is not identical with the antiscorbutic vitamin *C*. P. H. P.

Comparison of Certain Oxidising Enzymes of the Higher and Lower Plants. M. E. Robinson. (*Biochem. J.*, 1924, **18**, 543–548.)—A fundamental difference appears to exist between the oxidases of the higher plants and those of the basidiomycetes, in that those of the former which have been examined consist of three components, a catechol derivative and two enzymes, oxygenase and peroxidase, whilst the oxidase of the basidiomycetes has so far been separated into two constituents only—an enzyme-like peroxide and peroxidase. The experiments of Onslow (*Biochem. J*, 1920, **14**, 535) on certain phanerogam oxidases have been repeated and his results confirmed. In the case of the mangold, the oxygenase and peroxidase could be demonstrated, whilst the presence of the catechol derivative, though indubitable, was sometimes difficult to show. The mangold contains a substance which is probably of a pectic nature, and this may be in combination with the catechol derivative. Probably the small content of the catechol derivative in the mangold, and also in the potato, may be due to the fact that these plants have a number of cultivated varieties, the chemical compositions of which may vary from that of the original types. The work of Gallagher (*Biochem. J.*, 1923, **17**, 515) on extractable plant phosphatides has been repeated; its interpretation as regards the oxidase reaction is very fully discussed in the text. P. H. P.

Oxidising Enzymes. VII. The Oxygenase of the Higher Plants. M. W. Onslow. (*Biochem. J.*, 1924, **18**, 549.)—The author criticises a statement made by Gallagher (*Biochem. J.*, 1923, **17**, 515) that there is no definite evidence for the existence, in the higher plants, of an enzyme (oxygenase) which catalyses the formation of peroxide, and that the presence of an autoxidisable lecithin-like substance is the cause of the rapid blueing of guaiacum; such a compound has been extracted from the potato tuber and the mangold root. A thermo-labile catalyst can be demonstrated readily in the potato tuber, mangold root and many other plants, but not in those of which the tissues show no direct blueing. The crude autoxidisable lecithin-like substance can be obtained from the tissue of a plant, *e.g.* the turnip root; which shows no direct blueing of guaiacum, and it needs some time (5 to 19 days) to autoxidise before giving any blueing with guaiacum in presence of peroxidase, whereas in the plant tissues the reaction is usually almost instantaneous. Many substances have this power of autoxidation and after some time can act as peroxides. P .H. P.

Influence of Colloids on the Reductase Test. A. I. Virtanen. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 141–151.)—When methylene blue is added to milk, as in the reductase test, part is adsorbed by the colloids present and part is in true solution. The time of reduction might, therefore, be expected to be affected by the presence of electrolytes which would, of course, alter the physical state of the casein, or by the presence of added colloids which would absorb more of the dye, or by added water which would alter the ratio between the natural colloids and the water. Experiment shows, however, that there is no marked effect traceable to these causes; it appears that in the reduction of the colour the equilibrium is so readily adjusted between the two phases that the influence of these factors is negligible. Barthel's method of applying the test was used, and there was no effect produced by the addition of either salts, or colloids, or water.
H. E. C.

Insulin from the Cod Fish. Direct Application of Picric Acid to the Islet Tissue. H. W. Dudley. (*Biochem. J.*, 1924, **18**, 665–668.)—A convenient method of extracting insulin from the islet tissue of fish is described. It consists in applying picric acid directly to the tissue, extracting the insulin-containing picrate fraction with watery acetone, and converting the picrate into a soluble hydrochloride. Even with material of less than perfect freshness, preservation in watery picric acid was successful. There is a large principal islet in the cod occurring as a cap over the top of the gall bladder, and it can be detached easily. A yield of 13·12 rabbit units per grm. of tissue was obtained from the islet tissue of the cod, although collected from fish which had been caught at varying intervals up to 24 hours previously. Probably more would be obtained from absolutely fresh islets. The islet tissue of the cod contains, weight for weight, apparently about ten times as much insulin as mammalian pancreas. P. H. P.

Note on the Estimation of Phosphorus in Blood. M. Martland and R. Robison. (*Biochem. J.*, 1924, **18**, 765–768.)—There are many pitfalls in the estimation of phosphorus compounds in blood, and although under normal conditions the consequent errors may not be very large, they may be considerably increased if the conditions are but slightly changed. Although not mentioned before, in the Bell-Doisy method (*J. Biol. Chem.*, 1920, **44**, 55) the intensity of the final colour varies considerably with changes in the degree of acidity of the solution in the first stage of the process. This may cause errors in the estimation of the total phosphorus after acid ignition. In estimating the total phosphorus by the modified method of Briggs (*J. Biol. Chem.*, 1922, **53**, 13), after acid ignition the total acidity comes near or above the upper limit of safety; hence the authors use ammonium molybdate solution without acid, but the usual Briggs molybdate solution for the standards. Losses traced to over-ignition were entirely avoided by using more moderate temperatures and stopping the ignition as soon as the white fumes of sulphur trioxide appeared. Trichloracetic acid produces a blue colour with the Briggs reagents in complete absence of inorganic phosphate, but it is produced slowly; hence, to avoid error, the colorimetric readings must be made not more than 30 minutes after adding the reagents. If, in estimating the inorganic phosphates in blood, even a few minutes are allowed to elapse between laking and precipitation of proteins, the results will be too high. The amount of inorganic phosphate in whole blood is increased after ether anæsthesia, and also as the result of shock consequent upon cardiac puncture. P. H. P.

Effect of Oxygen Supply on the Metabolism of *Bacillus Coli Communis*. M. Stephenson and M. D. Whetham. (*Biochem. J.*, 1924, **18**, 498–506.)—A technique previously applied to the study of the metabolism of a non-fermenting bacillus was extended to one of a fermenting type (one which breaks down carbohydrates and other compounds in such a way that intermediate products collect in the medium), *B. coli* being employed. Three strains of it gave similar results. Details of the work are given, and curves showing the results. The carbon dioxide excretion and oxygen consumption of *B. coli* on a glucose inorganic medium were followed. The organism was almost anaerobic during the early stages of glucose decomposition. In an atmosphere of oxygen there was an increase in carbon dioxide excretion and a larger increase in oxygen consumption. This did not correspond, in the early stages, to an increased utilisation of glucose. Metabolism continued longer in oxygen than in air, the limiting P_H not being reached so soon. The increase of gaseous exchange with increase of oxygen pressure was attributed to an effect on some acid decomposition product of glucose. Growth on lactic acid (in the form of the ammonium salt) was studied in air and oxygen. Increased oxygen tension greatly increased the disappearance of lactic acid and production of carbon dioxide. Growth did not occur on lactic acid in anaerobic conditions. Succinic and acetic acids and glycerol were also found to be suitable sources of carbon for *B. coli* in air, but not in anaerobic conditions. A possible explanation of these facts may be found in the energy relations of the reactions involved.

P. H. P.

Proteolytic Action of *Bacillus Granulobacter Pectinovorum* and its Effect on the Hydrogen Ion Concentration. W. H. Peterson, E. B. Fred and B. P. Domogalla. (*J. Amer. Chem. Soc.*, 1924, **46**, 2086–2090.)—*Bacillus granulobacter pectinovorum* causes a rapid hydrolysis of the proteins during the fermentation of maize mash; from 50 to 75 per cent. of the total protein is converted into soluble products in the course of the fermentation, which is usually complete in three to four days. One-half of the soluble products may be formed within twenty-four hours. The hydrolysis results chiefly in the formation of simple peptides and amino acids; due to these buffers and to acids of low dissociation, a high titratable acidity may be produced without causing much change in the hydrogen ion concentration. W. P. S.

Toxicological and Forensic.

Circulation of some Heavy Metals in the Organism (Mercury, Bismuth and Lead). S. Lomholt. (*Biochem. J.*, 1924, **18**, 693–711.)—These experiments were carried out on rabbits. The metal compounds to be investigated were injected at short intervals into different portions of the muscles of a rabbit's limbs, and, after 10–20 days, the animal was killed under a narcotic by bleeding. The principal organs were then removed, together with a sample of the blood, for examination. The material analysed comprised: (1) The daily eliminated amount of urine and of fæces; (2) parts of muscles containing the remaining deposits of the metal compounds injected; (3) the most important viscera; (4) in some cases the total remaining parts of the rabbit's body. The results are given, partly in numbered columns, partly—to simplify the survey—in a series of diagrams. Four mercury compounds were investigated, mercurium benzoicum, acidum mercurisalicylicum, calomel, and metallic mercury finely divided in oil, by means of the method of Lomholt and Christiansen (*Biochem. Z.*, 1917, **81**, 356), which comprised destruction of organic substances, precipitation with hydrogen sulphide, solution of the precipitate, and electrolysis on thin gold electrodes which were weighed on a Nernst micro-balance. A large proportion of mercury was eliminated in the urine. In percentage the kidneys are the organs by far the richest in the metal; the liver comes second. The percentage distribution of the mercury in the organism is almost uniform with these four compounds, but the actual amount of mercury found is proportionately greater with the quickly absorbed preparations (mercurium benzoicum and acidum mercurisalicylicum) than with calomel, and especially with metallic mercury.

Amounts of bismuth were estimated by the radio-chemical method of Hevesy and Paneth (Aston: The Isotopes, 1922, Cambridge, 1924). Small quantities of radium E were mixed with the inactive bismuth to be used. When the radio-activity of the mixture was known, the bismuth was estimated by determining the β-radiation of the radium E. [Christiansen, Hevesy and Lomholt (*Compt. rend.*, 1924, **178**, 1324)]. Bismuth quinine iodide (Viochine) and bismuth hydroxide were examined. With the latter a little lampblack was added to the oil suspension, as it was difficult to find and excise the parts where the injections had taken place. The greatest amount was found in the kidneys, not only relatively, but positively, twice as much being in the kidneys as in the liver. Bismuth and mercury compounds resemble each other as regards elimination and circulation, and are alike in other points, as they cause the same symptoms of poisoning (nephritis, stomatitis and colitis) and possess the same peculiar effect on syphilis.

The analytical method for lead was analogous to that used for bismuth, except that radium D was used instead of radium E [Hevesy (*Biochem. J.*, 1923, **17**, 439)]. Lead hydroxide was used, and lampblack was added to the emulsion. About twice as much of the metal was found in the liver as in the kidneys. Therefore the three metals investigated showed much the same conditions in the organism, but whereas with bismuth the kidneys (and the urine) play the most prominent rôle, with lead the liver (and the fæces) seem to be just as important. Mercury comes between the two, somewhat nearer to bismuth. P. H. P.

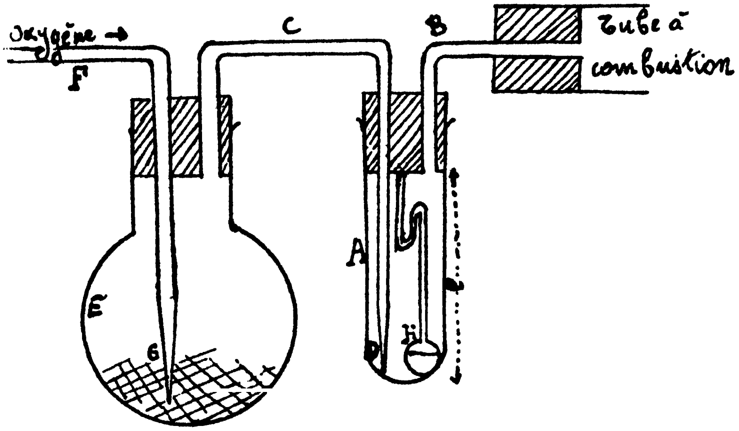
Agricultural Analysis.

Effects of the Method of Desiccation on the Nitrogenous Constituents of Plant Tissue. K. P. Link and E. R. Schule. (*J. Amer. Chem. Soc.*, 1924, **46**, 2044–2050.)—Experiments with beet leaves, maize leaves, maize ears, and common barberry leaves showed that drying at different temperatures had no effect on the total nitrogen content of the tissues. Drying at 80° and 90° C. caused coagulation of the soluble nitrogenous constituents in all cases. When the materials were dried at 32° and 54° C. proteolytic decomposition occurred, but in some instances this effect appeared to be counterbalanced by coagulation of soluble nitrogenous constituents. The general effect of all the methods of desiccation was a decrease in the amount of soluble nitrogen, due to coagulation; at 65° C. this was the only significant alteration. W. P. S.

Organic Analysis.

Organic Analysis by a Volumetric Method. J. Lindner. (*Chem. Zeit.*, 1924, **121**, 725.)—The usual method of combustion is employed, but connected with the combustion tube is an apparatus in which the products of combustion are brought into intimate contact with a halogen-phosphorus organic derivative, the compound $C_{10}H_7POCl_2$ being generally used. The water vapour produced during combustion is transformed by this compound into hydrochloric acid, which is absorbed by a small quantity of water and is readily estimated by titration. The carbon dioxide remaining in the products of combustion is absorbed by 0·1 *N* barium hydroxide solution and is estimated by back-titration. The analysis can be carried out on 20 mgrms. of the original substance and gives an accuracy of 0·05 per cent. in the case of the hydrogen, and of 0·1 per cent. in the case of the carbon. R. F. I.

Method of Analysis of Volatile Organic Substances by Combustion in an Open Tube. E. Carriere and C. Leenhardt. (*Bull. Soc. Chim.*, 1924, **35**, 1206–1207.)—By means of the device described a volatile liquid to be analysed by combustion is caused to evaporate regularly and is prevented from condensing in the front portion of the combustion tube. The substance is weighed in a stout bulb with a thin, sealed vertical neck having an S-bend. This is placed in a test-tube connected with the combustion tube by a tube flush with the bottom of the stopper. Through the latter also a tube, drawn out at its lower end, passes to the bottom of the test-tube, its other end being flush with the bottom of the stopper of a small round-bottomed flask. Into this flask oxygen is passed through a tube reaching the bottom of the flask and with its drawn-out end below a quantity of broken glass. The oxygen is warmed in this flask and then passes into the test-tube and causes gradual evaporation of the liquid from the bulb, the end of the neck of which is broken by forcing in the stopper of the test-tube. The last traces of the liquid are eliminated by subjecting the test-tube to alternate heating and cooling. T. H. P.



New Reaction of Aldehydes. R. De Fazi. (*Gazz. Chim. Ital.*, 1924, **54**, 658–667.)—The unsaturated compounds formed by condensation of fluorene with piperonaldehyde, cuminaldehyde, *m*-tolualdehyde, cinnamaldehyde, *p*-dimethylaminobenzaldehyde, and furfuraldehyde give various colorations with concentrated sulphuric acid (*cf.* ANALYST, 1921, **46**, 376), but this is not the case with the compounds formed by reduction of these condensation products. Such products are not formed by acenaphthene under the conditions employed, but acenaphthenone reacts with cyclic aldehydes in presence of sodium hydroxide, giving compounds which are coloured violet-red by concentrated sulphuric acid. T. H. P.

Colour Reaction Common to Formaldehyde and Glyoxylic Acid. Fosse and A. Hieulle. (*Comptes rend.*, 1924, **179**, 636–638.)—Like formaldehyde, glyoxylic acid gives a deep magenta coloration with Schryver's reagent, composed of 2 c.c. of 1 per cent. phenylhydrazine hydrochloride solution, 1 c.c. of 5 per cent.

potassium ferricyanide solution, and 5 c.c. of concentrated hydrochloric acid. The reaction is detectable with 0·001 mgrm. of glyoxylic acid at the concentration 1:1,000,000. If the coloured liquid is shaken with ether, the ether extracts the base derived by the dissociation of the colouring matter and regenerates the latter if treated with a few drops of hydrochloric acid. T. H. P.

Exact Methods for the Analysis of Gaseous Fuels. E. Ott. (*Helv. Chim. Acta*, 1924, **7**, 886–898.)—The author, with H. Deringer, has investigated the different absorbents for gases occurring in illuminating or fuel gas. It is shown that fuming sulphuric acid is preferable to bromine water for the estimation of heavy hydrocarbons, as it is not only easier to handle but more accurate, especially for benzene or ethylene. Fixed pipettes loaded with glass rod are better than the ordinary moveable type. When only traces of ethylene or benzene are present and have to be estimated it is advisable first to remove any oxygen by means of phosphorus, and carbon monoxide by copper ammonium chloride. A summary of previous work on the same subject is given. H. E. C.

Examination of Commercial Pinene. A. Gawalowski. (*Zeitsch. anal. Chem.*, 1924, **64**, 471–473.)—The method of testing pinene by means of strong hydrochloric acid (*Zeitsch. anal. Chem.*, 1923, **63**, 121) may conveniently be carried out in a "pinenometer," a stoppered graduated cylinder of 15 c.c. capacity. The zero (top) mark corresponds to 15 c.c., the 50 mark to 10, and the 100 mark to 5 c.c. volume. The treatment of the sample of pinene is carried out as previously described; the volume of the top layer is read off after 24 hours; the figure multiplied by 0·7884 gives the percentage of pinene *plus* camphene. For the detection of chlorine, a flattened platinum wire is sprinkled with copper oxide, ignited, impregnated with the material, and again ignited. When the wire becomes incandescent after combustion of the organic matter, the flame is coloured green if chlorine is present. An approximate estimation of the chlorine content is possible if the persistence of the green coloration is measured in seconds and compared with that of an equal weight of a standard preparation of known chlorine content.

W. R. S.

Estimation of Thymol and Carvacrol in Thyme Oils. C. E. Sage and W. G. Dalton. (*Perfumery and Essential Oil Record*, 1924, 345.)—The oil under examination is shaken with a 4 per cent. solution of sodium hydroxide in a boiling water bath and washed with water. The alkaline solution is acidified, and the liberated phenols extracted with petroleum spirit. This extract is dried with anhydrous sodium sulphate and allowed to evaporate in an open dish, complete removal of the petroleum spirit being effected by warming the residue to 35° C. for a few minutes. The solidification point of the mixed phenols is determined, and an allowance is made for the traces of the non-phenolic constituents of the oil also present. For this purpose a range of mixtures was prepared of pure thymol and pure carvacrol with non-phenolic constituents of the oil, the mixed phenols were prepared from these by the above method and their solidification points determined and plotted on a curve. By comparing the freezing points of the mixed phenols of the oil under examination with the points on the curve the percentage of thymol can be estimated. The solidification point of thymol is given as 49° C. and that of carvacrol as 0° C. R. F. I.

Methylene Blue as an Indicator of Bleaching Damage. E. Ristenpart. (*Cellulosechem.*, 1924, **5**, 8; *J. Textile Inst.*, 1924, **15**, 254A.)—The fabric to be tested is dyed by immersing 5 sq. cm. of it in 500 c.c. of a cold 0·001 per cent. solution of methylene blue for 10 min. It is then rinsed, dried in contact with a smooth surface, and the "degree of white" is measured in terms of Ostwald's notation (*cf.* Abst., p. 607). In this, a matt surface of barium sulphate is taken as a standard, and between this and black are 100 degrees. Damage by bleaching is indicated by increased affinity for methylene blue, and a comparison may be obtained in the form of a ratio, Q, between the "degree of white" of the original sample and the "degree of white" of the sample dyed with methylene blue. If this ratio be Q in the bleached goods and Qu in the unbleached, then the "methylene blue value" is expressed as the difference produced by bleaching,

$$M = (Q - Qu)/Qu.$$

In hypochlorite bleaching the "methylene blue value" increases with the concentration of the chlorine, and also with the rise in temperature, but not with the alkalinity. A permanganate bleach produces far more oxycellulose than a hypochlorite bleach containing the same amount of active oxygen. R. F. I.

Inorganic Analysis.

Separation of Mercury from Arsenic. P. Wenger and M. Schilt. (*Helv. Chim. Acta*, 1924, **7**, 907–909.)—An accurate separation of mercury from arsenic may be effected by means of ammonia or sodium bicarbonate. The mixed sulphides are washed with cold ammonium hydroxide solution, which should also contain ammonium chloride in order to prevent the formation of any colloidal mercury sulphide, and then with ammonium chloride solution until the washings are neutral. Alternatively, sodium bicarbonate may be used, but in this case the solution must be warm to ensure complete solution of the arsenic sulphide. For the subsequent estimation of the mercury the sulphide is collected on a tared Gooch crucible, dried at 105° to 120° C., then washed with ammonium chloride solution and three times with alcohol, extracted with carbon disulphide to remove sulphur, washed again with alcohol and finally with ether, then dried and weighed.

H. E. C.

Electrolytic Separation of Copper, Antimony and Bismuth from Lead. A. Lassieur. (*Comptes rend.*, 1924, **179**, 632–634.)—In the electrolytic separation of lead from copper, antimony and bismuth in presence of a reducing agent such as hydroxylamine, an acidity corresponding with at least 15 to 20 per cent. of hydrochloric acid (1·19) is necessary. To obtain a quantitative lead deposit with the apparatus described by the author (*Comptes rend.*, 1923, **177**, 1114), the auxiliary potential should be 440 millivolts, the volume of the electrolyte being 200 c.c. and the temperature 50° to 60° C.; from 1 to 2 grms. of hydroxylamine hydrochloride are sufficient. Hydrofluoric acid, the preliminary use of which is sometimes necessary, may be present in the liquid in the proportion of 2·5 per cent. of the concentrated acid in place of some of the hydrochloric acid, the auxiliary potential being lowered in this case to 400 millivolts; glass vessels are still usable under such conditions.

In presence of lead, bismuth deposits well from a solution containing nitric acid, either alone or together with hydrochloric acid, and hydroxylamine hydrochloride, the auxiliary potential being from 200 to 240 millivolts; the lead forms a good cathodic deposit from a nitric acid solution containing hydroxylamine. The separation of copper and lead is easily effected in hydrochloric acid solution containing hydroxylamine hydrochloride, the copper being completely deposited at an auxiliary potential of 240 millivolts.

In separating antimony from lead, the antimony forms a black, loose coating if deposited from a solution of its trichloride in hydrochloric acid containing hydroxylamine, but a good, complete deposit is obtained from the antimonic salt with the auxiliary potential 240 millivolts. The lead is deposited on a cathode either of platinum or coated with antimony with the auxiliary potential 350 millivolts, but the concentration of the lead must not exceed 0·2 grm. per 100 c.c., since otherwise antimony becomes entangled with the lead. T. H. P.

Detection and Estimation of Traces of Calcium and Magnesium. F. Pavelka. (*Chem. Zeit.*, 1924, **121**, 728.)—Calcium and magnesium salts can be detected in alcoholic solution by means of ammonium ferrocyanide, which causes the formation of a finely crystalline precipitate. In dilute solution, *e.g* in water analysis, a permanent cloudiness is produced, and this effect may be made the basis of a nephelometric method of estimation. This test is claimed to be very much more sensitive than those with any of the usual reagents.

R. F. I.

Separation of Yttrium from the other Elements of Gadolinite. L. Rolla, V. Cuttica and L. Fernandes. (*Gazz. Chim. Ital.*, 1924, **54**, 617–622.)—Yttrium may be separated from the other elements occurring with it in gadolinite by fractional crystallisation of the mixed sulphates formed with thallous sulphate, the solubility being lowest for the yttrium double sulphate. These double sulphates are stable compounds and are more soluble at low than at high temperatures.

T. H. P.

Direct Nesslerisation Micro-Kjeldahl Method and a Modification of the Nessler-Folin Reagent for Ammonia. F. C. Koch and T. L. McMeekin. (*J. Amer. Chem. Soc.*, 1923, **46**, 2066–2069.)—The substance is heated with concentrated sulphuric acid until dissolved, the mixture is cooled, a small quantity of 30 per cent. hydrogen peroxide is added, and the heating is continued. The addition of the peroxide causes very rapid oxidation of the carbonaceous matter present, and there is no loss of ammonia. The use of hydrogen peroxide in the ordinary Kjeldahl method is also advantageous in the case of substances containing a high proportion of carbohydrates and fats, since it prevents troublesome foaming. The modified Nessler-Folin reagent is prepared by dissolving 22·5 grms. of iodine in 20 c.c. of water containing 30 grms. of potassium iodide; 30 grms. of metallic mercury are then added, and the mixture is shaken and cooled until all yellow colour due to iodine has disappeared. The solution is then decanted and concentrated iodine solution is added, drop by drop, until a drop of the mixture gives a reaction with starch solution. The whole is then diluted to 200 c.c. and mixed with 975 c.c. of 10 per cent. sodium hydroxide solution. W. P. S.

Volumetric Estimation of Ammonium Salts. S. Lövgren. (*Zeitsch. anal. Chem.*, 1924, **64**, 457–470.)—The direct titration of ammonium salts at ordinary temperature by means of caustic alkali was investigated, thymolphthalein being used as indicator. The end-point (colourless to blue) was found to be sufficiently sharp in alcoholic solution, provided the final alcohol concentration was at least 50 per cent. The final volume should not much exceed 10 c.c., otherwise the sharpness of the end-point is impaired; one c.c. of a saturated solution of thymolphthalein in 50 per cent. alcohol is added. The maximum error is stated to be ± 1 per cent. The strength of the standard alkali solution should be chosen with a view to preventing undue dilution; with a 0·1 *N* solution it is unsafe to titrate more than 0·01 grm. of ammonium chloride. For larger quantities, *e.g.* 0·3 grm., the salt is dissolved in 2 c.c. of water, with addition of 7 c.c. of alcohol and 1 c.c. of indicator, and titrated with *N* sodium hydroxide to the appearance of a blue colour. The use of a standard ammonium salt solution is recommended.

W. R. S.

Volumetric Estimation of Hydrazine by the Iodine, Bromate, Iodate, and Permanganate Methods. I. M. Kolthoff. (*J. Amer. Chem. Soc.*, 1924, **46**, 2009–2106.)—Under proper conditions, which are given below, all four methods yield accurate results. *Iodine Method.*—Twenty-five c.c. of approximately 0·1 *N* hydrazine sulphate solution are treated with 1 grm. of sodium hydrogen carbonate, and the solution is titrated with 0·1 *N* iodine solution until the yellow colour persists for two minutes; the last few drops of iodine solution required should be added slowly. *Bromate Method.*—The hydrazine sulphate solution is mixed with an equal volume of hydrochloric acid (sp. gr. 1·19), a few drops of indigo or methyl-red solution are added, and the mixture is titrated with 0·1 *N* potassium bromate solution until the colour disappears. When indigo is used, an excess of 0·14 c.c. of bromate solution is required; with methyl-red, the excess is 0·22 c.c. at the end-point. These amounts must be subtracted as a correction. *Iodate Method.*—Six c.c. of carbon tetrachloride and at least 25 c.c. of hydrochloric acid (sp. gr. 1·19) are added to 25 c.c. of hydrazine sulphate solution in a stoppered flask, and the mixture is titrated with standard iodate solution until the carbon tetrachloride layer is decolorised after the mixture has been shaken vigorously: $N_2H_4 + KIO + 2HCl = KCl + ICl + N + 3H_2O$. *Permanganate Method. A. In acid solution.*—Ten c.c. of 4 *N* hydrochloric acid are added to 25 c.c. of hydrazine sulphate solution, the mixture is heated to boiling and titrated with 0·1 *N* permanganate solution until a pink coloration is obtained; this colour disappears after a time *B. In alkaline solution.*—Twenty c.c. of hydrazine sulphate solution are mixed with 50 c.c. of 0·1 *N* permanganate solution and 10 c.c. of 4 *N* sodium hydroxide solution; after thirty minutes, 1·5 grm. of potassium iodide and 20 c.c. of 4 *N* sulphuric acid are added, and the liberated iodine is titrated with 0·1 N thiosulphate solution. In both cases the hydrazine is oxidised according to the equation:

$$N_2H_4 + O_2 = N_2 + 2H_2O.$$

W. P. S.

ERRATUM.

Separation of Rhodium from Platinum:—On page 545, line 33, read "*not* more than 1 grm."

Physical Methods, Apparatus, etc.

Colloidal Poles for observing the Emission Spectra of Solutions. J. Errara. (*Bull. Soc. Chim. Belg.*, 1924, **33**, 449–451.)—As has been shown by Gramont, the wave length of the ultimate rays of spark spectra of dilute solutions affords a ready means for the detection of metals. The spectra are obtained by sparking the solution between poles of gelose (an agar-agar product) at the secondary terminals of an induction coil. To prepare the poles the gelose should be washed for a week in slowly running distilled water, then dried by pressure to a water content of about 4 per cent., moulded into pencils and baked in an autoclave at 120° C. for 10 minutes. The pencils are cut into pieces about 7 by 50 mm., which are soaked for a few minutes in the solution to be tested, and used as poles for the sparking. The following rays are due to the gelose alone, the three rays of calcium and magnesium, one of sodium (3303), two of silver and copper, and 3064, 3067, 3089 due to water, and to air 3159, 3371, 3536, 3577.

Element.	Limit of concentration for μ_1.	Ultimate rays.
Silver	1/1000000	3280 $7\mu_1$; 3383, μ_2; 2437 μ_3
Cadmium	1/1000000	2288 μ_1;
Mercury	4·32/10000	2536 $5\mu_1$; 3650 μ_2; 4046 $6\mu_3$
Calcium	—	3933 $7\mu_1$; 3968 $5\mu_2$; 4226 $7\mu_3$
Magnesium	—	2852 $1\mu_1$; 2795 $5\mu_2$; 2882 $7\mu_3$
Copper	—	3247 $5\mu_1$; 3274 μ_2.

H. E. C.

Differentiation of Natural and Culture Pearls. A. Dauvillier. (*Comptes rend.*, **179**, 818–819.)—A method of distinguishing between natural and culture pearls has been based on the photographic examination of the figures of Laue or the diffraction rings produced when they are traversed axially by monochromatic X-rays. For this purpose the object is exposed for some hours to the K rays of rhodium and of silver produced by means of the quartz tube previously described (*Comptes rend.*, 1921, **172**, 1915), and the photographic plate is placed at a distance of 12 cm. while an intensifying screen of calcium tungstate is used. In the case of natural pearls the photograph shows a system of regular rings due to the doublets Kα and the rays Kβ. Pearl nacre, on the other hand, invariably gives more or less regular Laue figures composed of more diffused stains than crystals produce. These stains tend towards a hexagonal system when the irradiation is effected normally to the "plane of cleavage." In the perpendicular direction they are more numerous, and show quaternary symmetry. Thus culture pearls give at the same time both the system of rings characteristic of the true pearl and stains which reveal the internal nucleus of nacre.

Colour Measurement. O. Meissner. (*Zeitsch. Physik.*, 1924, **21** 68–72; *J. Textile Inst.*, 1924, **15**, 230A.)—In Ostwald's colour scheme (*cf. J. Soc. Chem. Ind.*, 1919, **38** 914A) each colour is defined by three characteristics, *viz.* the colour tone, c (in the scale of 100 divisions, 0 = yellow, 25 = red, 50 = blue, etc.), the proportion of white, w, and the proportion of black, s. From these the purity of the colour, r, is derived—$r = 100 - (w + s)$. The grey content is $w(w + s)$. All derivatives of a definite pure colour form a colour triangle, and by its rotation about the grey axis there arises a double cone, the properties of which are mathematically described; by means of it the characteristics of a mixed colour are determined from its components. It is shown that the purity of a composite colour is always lower than the arithmetical mean of the purities of the separate shades. An example is given of the formation of grey from three colours.

Reviews.

KONDUKTOMETRISCHE TITRATIONEN. By DR. I. M. KOLTHOFF. Pp. viii, +94. Dresden and Leipzig: Theodor Steinkopff. 1923. Price 2s. 9d. (paper cover).

This short monograph gives a clear and concise account of the various ways in which the measurement of conductivity can be applied to the solution of analytical problems. The first chapter deals generally with electrolytic dissociation, the hydrolysis of salts, the formation of complex molecules, etc., and in the second and third chapters the theoretical and practical considerations involved in conductivity measurements are discussed. In succeeding chapters the application of the method to analytical problems is considered and diagrams are given of the various graphs obtained in titrating acids and bases of various strengths and in titrations involving precipitations. In the greater number of cases the conductivity graphs consist of straight lines which intersect at the points at which the reaction is complete. Accurate results may be obtained in the titration of weak acids with weak bases in cases where ordinary indicators give an uncertain colour change. The method may be used with highly coloured liquids and is of considerable practical value in the examination of wines, vinegar and possibly beer. It is by no means generally applicable, as in some cases the phenomena of dissociation and hydrolysis combine to produce a graph which is more or less uniformly curved, and in which no sharp indication of the completion of a reaction can be observed. In many natural products, such as beer and fruit juices, the significance of the data given by the conductivity method is not clear, and further research is needed. From a consideration of the many examples given the impression is gained that titration by conductivity methods is not likely to be of much value in the ordinary analytical laboratory. It is rather in the technical examination of a large number of samples of a particular product, such as wine, that it may often afford information which it would otherwise be difficult or even impossible to obtain. G. W. MONIER-WILLIAMS.

ANALYTICAL CHEMISTRY, Vol. II., QUANTITATIVE. By F. P. TREADWELL and W. T. HALL. Sixth Edition. Pp. xiii.+811. New York: Wiley & Sons; London: Chapman & Hall. 1924. Price 25s. net.

This well known work has been completely revised, but, as a comparison with the previous edition shows, the revision has been mainly textual. A few fresh methods have been included, and some of the old methods have been modified, apparently in accordance with the procedures adopted by the Bureau of Standards and by other American testing bodies. The standards of mass and volume now introduced into the book are those of the Bureau of Standards—so much so that the term millilitre (ml.) has been substituted for the more familiar cubic centimetre. The introductory section has been enlarged to include some treatment of topics such as the calculation of the precision of analytical data, adsorption by precipitates, the care of platinum, and sampling. Fuller consideration is given throughout the book to theoretical principles, *e.g.* the practical importance of a knowledge of the magnitude of the solubility products of certain sulphides, discussed on pp. 161–3. Fundamental analytical procedures also receive increased attention, in particular, the preparation of substances for analysis.

The treatment accorded to Indicators in the earlier editions was far from satisfactory, and an attempt has been made in the present issue to remedy this, by the inclusion of a brief study of Hydrogen Ion Concentrations, together with a description of Hildebrand's method of carrying out electrometric titrations with the use of the hydrogen electrode. The method is erroneously ascribed to Hillebrand. It is felt that the value of this knowledge from the standpoint of volumetric analysis has not been rendered sufficiently clear, and this is especially the case in its application to the choice of indicators. Thus typical neutralisation curves of various acids and bases would have been invaluable to the student in his selection of suitable indicators, and so would have been the electrometric titration curves of Hildebrand to explain the use of hydroxy compounds, *e.g.* mannitol, glycerol, in the titration of boric acid in presence of phenolphthalein. Küster's method of preparing alkali free from carbonate is still retained, but no mention is made of the recent and more convenient electrolytic method (*J. Amer. Chem. Soc.*, 1920, **42**, 488). Greater attention is given to the calibration of apparatus. The general treatment of gas analysis remains unchanged; the estimation of carbonyl sulphide has been added.

The volume cannot be regarded as exhaustive, for many established methods have not yet been included and the estimation of many elements receives either a cursory note or no mention at all. The work is, however, and in its revised form will remain, a reliable and valuable aid to the analyst and will continue to rank high among the few standard works on analytical chemistry.

HUBERT T. S. BRITTON.

MODERN CEREAL CHEMISTRY. D. W. KENT-JONES, B.Sc., F.I.C. Pp. 324. The Northern Publishing Co., Ltd. Price 25/- net.

This book, the author states in his preface, is written for cereal chemists and progressive millers. It deals with the chemical and physical properties of wheat, wheaten flour, and bread, with their physical and chemical examination, and with the operations of milling and baking. In general, it may be said that the book deals only with roller milling and white flour and bread; other flours are hardly touched upon, and the bye-products of milling are almost unnoticed, so that "cereal chemistry" is perhaps rather too wide a title; there is no doubt, however, that the "modern" is justified. It is not unfair to say that the book is a plea, and a very interesting plea, for white flour and for the bleaching and "improving" of white flour.

The author considers milling, flour itself, and the baking of bread largely from the view points of colloid chemistry and H-ion concentration, whilst the vitamin point of view is not neglected. Results of researches of recent years are presented as a consecutive story, full references being given to original papers.

In order to prepare the reader for consideration of the subject from these modern points of view, a separate chapter is devoted to the above-mentioned three subjects. H-ion concentration is clearly expounded and the necessary apparatus described. The chapter on colloidal chemistry is less successful and would be more helpful if the principles were more carefully considered, and their bearing upon the matter at issue, namely flour, were kept more in mind. It is true that a further chapter is devoted to the colloidal chemistry of flour, but this deals also very largely with H-ion concentration and diastatic power, so that the colloidal aspect gets confused. The chapter on vitamins is a digest of the first edition of the well known report of the Medical Research Committee on this subject.

What may, for the want of a better expression, be termed the "ordinary" analysis of flour occupies a subsidiary position in the book. The difficult question of the determination of moisture is fully and frankly treated. The fact that the figure for moisture content is almost meaningless unless the exact analytical procedure is given is fully brought out, and in this connection the progressive miller might well be pardoned if he doubted whether the cereal chemist knows what he is doing. A similar difficulty with regard to oil or fat determinations is referred to.

With regard to arsenic, the inclusion of a quantitative Reinsch process is surprising, and the use, in this test, of strips of copper 1 by 2½ cm. in area is to be deprecated for delicate work. A modification of the Gutzeit process, in which the hydrogen and arsine are bubbled through lead acetate solution and dried by calcium chloride, takes the place of the usual method. The determination of gas-production power and diastatic activity are included. An omission is that of a process for the determination of total phosphorus. The determination of starch by washing out, drying, and weighing, is simple, but does not seem to be a very refined method.

A prominent place is given to the question of bleaching and flour improvers. Here the author is far from impartial. This part of the book is largely an account of the patent case of 1909 (*Flour Oxidising Co.* v. *Hutchinson*), and of the action taken by the Hull Corporation with regard to persulphate in 1913. It is hardly correct to say with regard to bleaching (even with oxides of nitrogen) that "in England the matter was settled" by the former case, nor can it be agreed that as a result of the latter case "persulphates are the one class of improvers that have definitely received legal sanction"; it is true that others have not, but it is going too far to say that persulphates have. There are many curious, and very disputable, statements in this connection. After being assured that the question of bleaching by oxides of nitrogen is settled (in their favour) we are comforted by the statement that "these lower oxides of nitrogen are, on the whole, quite harmless non-corrosive gases." It is not easy to see what is meant by "new flour improvers should only be used publicly after the fullest of careful investigations." "Should this matter" of persulphates "or a similar one be opened again the views expressed by Mr. Jago may be taken almost as a standard," seems to be pressing standardisation too far. Not many Public Analysts will agree with the statement, flattering though it is, that "with the just and necessary strict Food and Drugs Acts which are in existence, and with the exacting and widespread examination of food by expert and skilful analysts all over the country, it need hardly be pointed out that the presence of a harmful flour improver, or one which would be likely to be injurious to the public health in the slightest degree, would not long survive." The whole treatment of these subjects is obviously too partial to be accepted without much reserve. It must be admitted, all the same, that the author makes out a very good case for improvers of one sort or another. In a few months' time we may all be wiser on these matters.

The suggested limit for calcium sulphate in acid phosphate is erroneously referred to in more than one place as a Board of Trade limit.

This book deals with matters of great national importance and of particular interest at the present time; it is full of enthusiasm for research, is never dull, and contains many valuable data. Its very modernity carries with it the drawback that in a short time, no doubt, many of the views expressed and quoted will have to be revised: it is to be hoped that the demand for this edition will render possible another in the not distant future. E. HINKS.

THE MANUFACTURE OF SULPHURIC ACID. CHAMBER PROCESS. By WILFRID WYLD. Pp. xii+424. London: Gurney & Jackson. 1924. Price 31s. 6d,

In this, the second volume of the revised edition of Lunge's "Manufacture of Acids and Alkalis," only the chamber process is considered. This avoids the somewhat fortuitous arrangement of the last edition, and enables the author to include new matter and to discuss it adequately.

The first part gives a comprehensive description of the construction and erection of lead chambers in general, followed by details of the various types, such as the Mills Packard, Moritz, etc., and also the Opl and other systems, in which towers to replace or work in conjunction with chambers are used. The various methods of supplying water to, and draughting the chambers are given, together with figures of the various sprays and apparatus used for these purposes, although, in many cases, these are rather what are to be found in manufacturers' catalogues than in actual practice. The same applies to the description of acid-elevating plant.

The use of pumps is rapidly coming into general use now that they can be constructed without glands or packing, and it would have been useful had data been given as to the life of these and their behaviour after being in use for some time.

The design and working of the Glover and Gay Lussac towers are well described, and figures are given as to the cubic capacity and performance of several towers.

Probably the most useful part of the book is Chapter III., which discusses in detail the supply of water and nitre to the chambers and the various factors which cause, respectively, improper or correct working of the chamber. The causes and methods of rectifying various plant troubles are fully described, and in this connection stress is laid on the testing of the chamber exit gases. Methods are given for carrying this out and for estimating the content of nitrogen acids, sulphur acids, etc. The author recommends working with 9 per cent. of oxygen at the exit in the case of spent oxide, although Lunge (4th edition) and others use 6 to 8 per cent.

The older and more recent theories of the chamber process are surveyed, and the conclusion reached is that the principal reactions are:

$$1.\quad SO_2+NOOH+O=SO_2(OH)(ONO).$$

$$2.\quad SO_2(OH)(ONO)+H_2O=H_2SO_4+NOOH.$$

Whether N_2O_3 exists for an appreciable time as undecomposed vapour or not, is not a decisive point in this case, as the hydrates NOOH may be introduced, and as the components NO and NO_2 act in the same way as N_2O_3. The excess SO_2 in the first chambers also gives:

$$2SO_2(OH)(ONO)+SO_2+2H_2O=3H_2SO_4+2NO.$$

A chapter is given on the purification of sulphuric acid, and other methods of producing it are briefly mentioned, *e.g.* from sulphates, bisulphates, etc.

Costs are not dealt with at length, owing to the great variations in prices during the last few years. References to patent and other literature are given throughout the text; hence older theories and systems are not included, which makes the book more valuable for practical use, although, in many cases, it would have been better if some mention had been made of the actual working success of many of the types of plant and systems mentioned. A fuller discussion on the merits or otherwise of intensive working would also have been advantageous.

The author is to be commended on having produced what is, in fact, a new treatise, rather than a revision of chamber acid processes.

ERIC A. REAVELL.

LIME AND MAGNESIA. By N. V. S. KNIBBS, B.Sc. Pp. xii+306. London: Ernest Benn Ltd. 1924. Price 30s. net.

This very useful book, if only because it brings together matter relating to the subjects of which it treats, which otherwise would be widely separated and not easy to refer to. The book is divided into three parts: Chemistry, Manufacture, and Uses.

In Part I., which perhaps may be looked on as the really distinctive and new feature of the work, after a chapter on the origin and occurrence of lime and magnesia compounds, there is one on the physical and chemical properties of lime, in which are collected data on specific gravity, optical, thermal, and electrical properties, solubilities, etc., of calcium oxide, hydroxide, and carbonate; and whilst these in themselves are very full, the value of the chapter is enhanced by the collection of references to authoritative and relevant literature at the end. This admirable practice is continued throughout the book. The following chapter deals with the chemical reactions of the same three substances, and is followed by two corresponding chapters dealing with magnesium oxide, hydroxide, and carbonate in a similar way. A short chapter on dolomite and magnesium limestones follows. The next is on the chemistry and physics of lime and magnesia burning; here are theoretical data on the dissociation of the carbonate by heat, discussion of experimental work on it, of over burning, of the effect of steam in calcination, etc.; and this is followed by a similar chapter on the hydration of lime and magnesia. These two chapters are perhaps the most important in the book, as affording a basis for placing the manufacture of quicklime and hydrated lime on a scientific foundation. The last chapter in this part deals with the analysis and testing of the materials and products of the lime and magnesia industries. The account given of the methods in common use is fairly satisfactory, though it is not easy to understand why some of these methods, which quite properly belong here, have been described in an appendix.

The second part contains a description of the plant used in the preparation of limestone for calcination, of the various forms of kiln, the control of the operation of burning, the calculation of the thermal efficiency of the process, and the methods of production of hydrated lime from quicklime. All this is gone into very fully, and appears, to one who is not a technologist in the industry, to be very well done, though in places it suggests compilation rather than first hand knowledge. The corresponding portion of the work on magnesia is very short, and in a future edition might with advantage have a little more space devoted to it.

The third part, after a consideration of the effect of impurities and of physical condition on the uses of lime and magnesia, proceeds to an enumeration and description of those uses. The uses of lime in agriculture, whether as quicklime, hydrated lime, or limestone, are very fully discussed, as are also the characteristics of the varieties of lime when used in constructional work, the setting of mortars, etc. The section on magnesia refractories and still more that on magnesia cement, are far from having the same fulness, and an extension of these would greatly improve the work.

The writing is clear, and the style interesting, and the printing and general get up of the work are admirable. Misprints and errors are few, though on page 184, line 5, the words "per lb." have intruded themselves unnecessarily and entirely changed the meaning. Some novelties in spelling disfigure the volume: "dessicator" is hardly a novelty perhaps, but "cintering" and "insipient" are distinctly so. Some technical writers, American especially, have so inured us to "more data *is* needed," that only those with weak nerves will suffer seriously from the shock of meeting it here; but when the author goes on to tell us of the actions effected by "a bacteria," even the strongest shudders. The use of the word "clog" in the sense of "clot," and of "insular" for "insulator," is to be deprecated; so is the habitual use in the text of formulæ as substitutes for the names of substances. But these, after all, are small blemishes, and do little to soil the virtues of a book which, in a sense, breaks new ground, and will be found of great use by many, chemists as well as lime burners. J. T. DUNN.

THE SYNTHESIS OF NITROGEN RING COMPOUNDS CONTAINING A SINGLE HETERO-ATOM [NITROGEN]. By CECIL HOLLINS, B.Sc., A.I.C. Pp. xiv.+423. London: Ernest Benn, Ltd. 1924. Price 55s.

Even in our age of specialisation it is, to some extent, surprising to find an author as well as a publisher for such a highly specialised book, and both the author and publisher are to be congratulated on the production of this valuable addition to chemical literature. It can confidently be assumed that Mr. Hollins' compendium will be of some considerable value, especially if one considers that it covers a field dealing with the chemistry of the pyrroles, indoles, quinolines, etc.

The book under review seems to offer a complete summary of the different methods used for the synthesis of the many compounds mentioned in it. It gives a detailed summary of the methods and it frequently also refers to the yields which have been obtained for some of the substances. Of interest is also the fact that

Mr. Hollins refers to cases in which generally successful reactions have failed to give the expected results. Such negative observations are of value, and it would be most useful if other writers would follow his example. Negative results are too often omitted from the literature of a subject, and this is obviously to be regretted. To the research chemist negative results are as important as the positive ones, and chemistry would gain much if more prominence were given to the former.

The most valuable part of a book of this kind is obviously the bibliography. The reviewer has therefore deemed it desirable to compare some 30 to 40 references with the original literature and he has found them to be correct in every respect. This certainly adds greatly to the value of the book. M. Nierenstein.

The Technique of Tissue Culture "in Vitro." Pp. 80. Price 7s. 6d.

Tissue Culture in Relation to Growth and Differentiation. Pp. 50. Price 5s. By T. S. P. Strangeways. Cambridge: Heffer & Sons. 1924.

These two volumes appear to be the first attempts to give in book form the technique of, and results achieved by, tissue culture in the laboratory. Although perhaps outside the usual run of books reviewed in this journal, it is by no means improbable that in future the analyst, among his many other activities, will be called upon to carry out experiments such as those described in the first of these volumes, when dealing with preservatives, toxicological investigations and related subjects.

The larger volume is devoted to detailed descriptions of apparatus and manipulations in the removal, culture and staining of animal tissues which the author has evidently written with meticulous care and a wide knowledge of the subject. The descriptions are perhaps unnecessarily detailed, since the worker likely to take up tissue culture will have no use for the instruction in elementary glass working on page 42, nor for the process of coating apparatus with paraffin wax, to which two and a half pages are devoted. Some confusion is caused by the frequent references to different sections of the book instead of to pages which are much more readily found, and the letters on Fig. 18 are so small as to be almost illegible.

The second volume provides brief but fascinating reading dealing with the observation of growth "*in vitro*," its relation to the physiology of the cell, modification of the developing cells by external agencies, such as the addition of various substances to the medium, variation of temperature, exposure to Röntgen rays, etc., and the relation of tissue culture to inflammation and tissue repair. The four collotype plates provided are excellent reproductions illustrating the stages of cell growth and mitosis, but unfortunately the magnification is not given, although this appears to be in the neighbourhood of ×500.

The author is to be congratulated upon the production of these two small volumes dealing with a subject which will undoubtedly become of increasing importance with the passage of time. T. J. Ward.

A

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THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held in the Chemical Society's Rooms, Burlington House, on Wednesday, 3rd December, 1924. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

A certificate was read for the first time in favour of Mr. Thomas Mann.

Certificates were read for the second time in favour of:—Messrs. Arthur Chapman Barnes, B.Sc., F.I.C., John Jacob Fox, D.Sc. (Lond.), F.I.C., Thomas Congalton Hay, and Harold Rupert Jensen, M.Sc. (Liv.), A.I.C.

The following were elected members of the Society:—Messrs. Arthur Cecil Brooks, William Donovan, Albert George Flower, Clifford Hollingsworth, George Marshall Norman, Charles Edward Sage, Percy Frank Spendlove, Charles William Spiers, Leslie Herbert Trace, and James Rawson Walmsley.

The following papers were read:—"The Analytical Value of the Melting Point of the Insoluble Volatile Acids from Fats," by G. Van B. Gilmour, B.Sc., A.I.C., A.R.C.Sc.I.; "The Use of Mitchell's Ferrous Tartrate Reagent in Qualitative Analysis," by A. H. Ware; "The Influence of Hydrogen Ion Concentration on the Colorimetric Determination of Pyrogallol and Catechol Derivatives," by S. Glasstone, M.Sc., Ph.D., F.I.C.; and "The Futility of Sealing: A Suggestion for its Safeguarding," by B. S. Evans, M.B.E., M.C., B.Sc., F.I.C.

Obituary.

ROBERT HELLON.

Robert Hellon was born at Workington in Cumberland, on September 13, 1854, and died on November 9, 1924. Some few years after his birth his father was appointed manager of the Bank of Whitehaven, and the family moved to that neighbouring town. Here, at a private school his education was begun, and it was finished at a school in Wrexham. Returning to Whitehaven, he studied analytical chemistry for a while with Dr. Tosh, and then proceeded to the School of Mines, where he came under the influence of Frankland and Percy. After taking his Associateship in 1873, he went to Heidelberg and gained all that working with Bunsen meant to the chemical student of those spacious days. Then, after studying in Hoffmann's laboratory in Berlin (1877), and with Würtz in Paris, Hellon took his degree of Ph.D., with distinction, at Heidelberg, early in 1878.

Returning to France, he joined Dr. Tcherniac, who had been his fellow student, and thence onwards was to be his life-long friend, and, as the result of this collaboration, thiocyanacetone was first prepared. The account of this work was published in the *Comptes rendus* of the Paris Academy of Sciences, and elsewhere.

He left France in 1883, and, entering into partnership with his friend, Alfred Adair, began to practise as an analytical chemist on the north-east coast. South Africa tempted Adair, however, and Dr. Hellon decided to try his luck alone in Whitehaven, where his parents were still living.

When, in 1889, the position of Public Analyst for Cumberland, Westmorland, and the Borough of Kendal became vacant, he was the successful candidate. His later appointments were as Agricultural Analyst for Cumberland and Carlisle and Public Analyst for Carlisle.

He was a pioneer in the application of chemistry to the leather industry, and was one of the founders of the International Association of Leather Trades Chemists.

He twice served on the Council of the Society of Public Analysts (1904–1905 and 1919–1920). He became an Associate of the Institute of Chemistry in 1878, and a Fellow in 1889.

In innumerable ways he did much for the spread of scientific education in the county of Cumberland. He was appointed Justice of the Peace in 1913.

From his wide experience in the field of chemistry and physics and his natural aptitude for, and deftness in, experimental work, he was particularly well equipped for the occupation that filled his days. Unfortunately his constant ill-health prevented him from communicating to his colleagues, save by word of mouth, the many ingenious methods of analysis he devised and brought to perfection.

The saddest day of his life was April 6, 1915, when his devoted wife died. Without children, and sometimes, during the long stretch of a dreary northern winter, confined for weeks together to the house with that distressing complaint, asthma, he was lonely indeed. At such times he lived largely in his private laboratory. Latterly, in the summer time, he took up botany, and became one of the leading authorities on the flora of his native county. When the failure of his eyesight and hearing became serious, wireless telephony came to him as a heaven-sent gift. As in everything he undertook, he threw his whole energy into his installation. He was ever grateful for this renewal of a contact with the outer world, which had been slipping away from him rapidly, and which he feared might cease almost completely.

The outstanding feature of his character was his gentleness. That must have been felt by everyone who came in contact with him. The writer has had the joy of his companionship for over thirty years, and he never heard him speak an unkind word to, or of, anyone. To the wayward he was ever generous. Children loved him, and it was his great delight to gather a turbulent band round his study fire and tell thrilling tales of adventure in the fairy fields of science.

He was what most of us wish to be, an able scholar and a very perfect gentleman.

THOMAS MANN.

On Certain New Methods for the Determination of Small Quantities of Arsenic, and Its Occurrence in Urine and in Fish.

BY H. E. COX, M.SC., PH.D. (LOND.), F.I.C.

(*Read at the Meeting, November* 5, 1924.)

So much has been written in the past on the determination of traces of arsenic that one would hesitate to offer any remarks on the subject were it not for the fact that some rather startling results have been reported by the late Prof. Bang to a Commission on Arsenical Poisoning in Sweden, and the conviction that the wet combustion process recommended by the Joint Committee of this Society and the Society of Chemical Industry in 1902 does not always give satisfactory results. Using new methods, the Swedish Commission reports the presence of quite considerable quantities of arsenic in the urine of normal persons, the amounts being such that in many cases chronic arsenical poisoning would be suspected. It has been generally assumed, without satisfactory reason, that urine is normally almost arsenic-free. In medical literature 0·03 mgrm. per litre is widely recognised as a maximum figure.

In the light of the results given by Bang (Arsenic Commission Bil. XI.) it appears that 60 per cent. of the population of Sweden could be suspected, on the above basis, of suffering from chronic arsenical poisoning. Thus, the following figures are given for 100 persons on a known hospital diet:

	As_4O_6 Mgrm. per litre.	Percentage of cases.
1.	0·00 - 0·01	21·1
2.	0·01 - 0·03	18·4
3.	0·04 - 0·06	23·7
4.	0·06 - 0·09	7·9
5.	0·10 - 0·20	21·1
6.	0·20 - 0·30	2·6
7.	0·30 - 0·70	5·2
		100·0

It will be appreciated how important such figures are in view of any criminal proceedings respecting arsenical poisoning. If 0·1 mgrm. or upwards of arsenious oxide were found in the urine, it would raise grave suspicion in the minds of chemists and physicians, yet it appears that seven times this amount may occasionally be present in the urine of a normal healthy person who is not taking arsenic in any form, except as a natural impurity in certain foods.

A search of the literature on this subject reveals that surprisingly few investigations have been made on normal urine, although there are plenty on real or supposed cases of chronic arsenical poisoning; also that in many instances the methods used have been, at best, only approximate. Thomson in 1905 (*Proc. Manc. Lit. Phil. Soc.*, 1905, **49**, 1) did, in fact, find from $\frac{1}{1000}$ to $\frac{1}{100}$ grain of arsenious oxide per gallon in a few samples from various places, and attributed the fact to arsenic in the air. Other workers, *e.g.* Sanger and Black (*J. Soc. Chem. Ind.*, 1907, 1123) have elaborated methods for the determination of arsenic in urine, but assumed it to be completely absent in the normal condition. Only last year Billeter and Marfurt (*Helv. Chim. Acta*, 1923, **6**, 780) stated that the average human body contains about 0·1 mgrm. arsenic as As_4O_6. This must be incorrect, as the urine alone may contain far more. It is considered by the Swedish Commission that the results obtained by earlier workers are low on account of the use of defective methods, and new processes are described. At the suggestion of some members of this Society I have carefully investigated these new processes and compared the results with those obtained by the older method.

Briefly put, my results confirm the main findings of the Swedish Commission, although their method of estimating the arsenic after the preliminary treatment is, in my opinion, open to criticism.

As the proposed methods, which really involve no new principle, are not available in English, French or German, they are abstracted here in some detail For the original papers I am indebted to Prof. Karl Petrén, of Lund; these were in the Swedish language, and my thanks are due to Mr. N. T. Foley, who was good enough to translate them for me.

A. BANG'S ORIGINAL METHOD.

(Bang and Bjork—Arsenic Commission Supplement VII.)

This method consists in the destruction of all organic matter by nitric and sulphuric acids by a modification of the Kjeldahl digestion; the arsenic is subsequently determined by distillation with hydrochloric acid and titration with dilute iodine solution.

To about 20 grms. of the substance to be tested are added 20 to 22 c.c. of sulphuric acid in a 300 c.c. Kjeldahl digestion flask, the mixture is warmed over a small flame, and concentrated nitric acid is added in drops, at the rate of about 8 per minute, by means of the apparatus shown (Fig. 1). Heating is continued until all organic matter is destroyed and a clear colourless liquid remains; the acid should fall directly on to the mixture, and the rate of heating should be so regulated as to avoid any large separation of carbon. When all the colour has disappeared strong heating is continued for 15 minutes, during which time all nitric acid is expelled. If any darkening appears more nitric acid must be added. After cooling, 50 c.c. of water are added, and the mixture is boiled, to remove nitrous

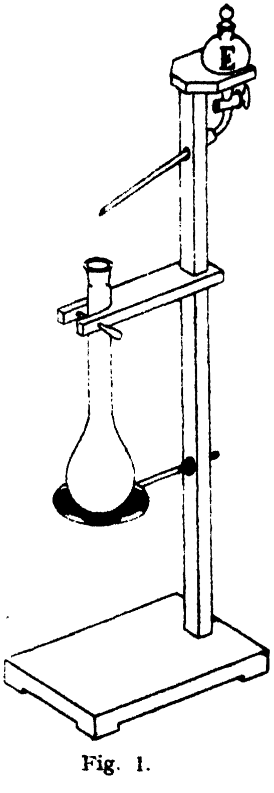


Fig. 1.

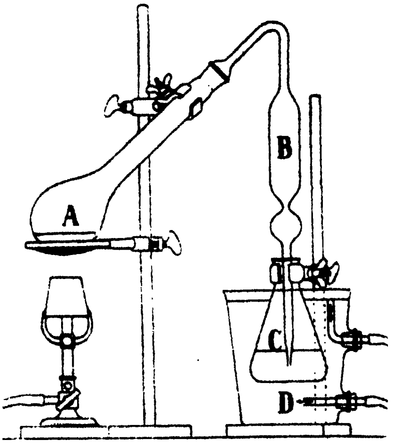


Fig. 2.

compounds, until white fumes again appear. A few crystals of potassium permanganate are now added, the colour of which should not be discharged. The volume of nitric acid added should be noted in order to apply the necessary corrections if it contains any perceptible amount of arsenic.

After completion of the combustion there are added 30 c.c. of water, 2 grms. of ferrous ammonium sulphate, 20 grms. of potassium chloride, and 2 grms. of potassium bromide. Then the flask is connected for distillation in the manner shown in the diagram (Fig. 2), except that the end of the tube does not touch the surface of the receiving liquor. The receiver contains 30 c.c. of 20 per cent. nitrite-free sodium hydroxide solution, 100 c.c. of water and 2 drops of phenolphthalein, and is cooled by running water. Distillation is continued until the colour of the alkaline solution becomes intensely red, then suddenly disappears; the acid distillate is neutralised by adding excess of sodium bicarbonate, and is then titrated with 0·005 *N* iodine solution.

It will be obvious that arsenic-free reagents must be used throughout; a blank test is made on the same quantities of acids and salts as are used in the determination, and the amount of iodine absorbed is deducted. It is not easy to obtain sufficiently pure reagents; the amounts employed are so large that serious error is easily introduced. Also, as is pointed out by Ramberg and Sjoström (Arsenic Comn. Supplement VIII.), there are errors inherent in the iodine titration due to absorption by substances other than arsenic. Boiling with water alone is not sufficient to remove all traces of nitric or nitrous acids, the complete elimination of which is a *sine qua non* of the process. Ramberg finds a preliminary treatment with dilute nitric acid always advantageous and sometimes essential; he removes nitrogen acids by ammonium oxalate and finally titrates with potassium bromate solution.

B. RAMBERG'S METHOD.

(This is the method finally adopted by the Commission in its Report).

A quantity of the substance which will contain not more than about 5 grms. of dry matter is introduced into a Kjeldahl flask and thoroughly wetted with about 15 c.c. of dilute nitric acid (sp. gr. 1·25). After a few minutes 22 to 23 c.c. of sulphuric acid are added, the mixture is heated, and concentrated nitric acid is dropped in, as in Bang's method. The atmosphere in the flask must at no time be free from red fumes. With substances difficult to burn the liquid is allowed to cool, 0·5 c.c. of nitric acid is added, and the flask re-heated; in this way the nitric acid is not vaporised too rapidly. When all organic matter is destroyed the addition of nitric acid is discontinued, and the heating continued until red fumes are no longer seen; then, after cooling, 25 c.c. of saturated ammonium oxalate solution are added and some fragments of glass, and the liquid is boiled until sulphuric acid fumes appear. After cooling, 20 c.c. of water are run in round the neck of the flask. Then there are added 50 c.c. of hydrochloric acid, 2 grms. of ferrous sulphate and 10 to 15 mgrms. of potassium bromide. If any yellow or brown colour appears at this stage, nitrogen acids are present and the experiment must be rejected. The flask is connected for distillation (the apparatus must have glass joints only), 150 c.c. of water are put into the receiver, and the exit tube made to dip 1 cm. below the surface. A volume of 20–25 c.c. is distilled in 10 minutes; to the distillate is added one drop of methyl-orange solution (1:5000), then it is slowly titrated at a temperature of 35°–40° with potassium bromate solution (0·1485 gr./litre; 1 c.c. = 0·20 mgrm. As_4O_6). The bromate solution is added a drop at a time towards the end of the titration, which is reached when the red colour of the methyl-orange is discharged; the end-point is quite sharp. A correction is applied for the blank on the reagents; this should not exceed 0·2 to 0·3 c.c.

Bang states that his method is accurate to 0·001 mgrm., that of Ramberg is put at 0·002 mgrm. For smaller quantities an electrolytic micro method is described, which is alleged to be accurate to 0·0001 mgrm.; in this case the acid solution containing the arsenic is introduced into an electrolytic Marsh apparatus of the type described by Trotman (*J. Soc. Chem. Ind.*, 1904, **23**, 177), and the mirror obtained is dissolved in 2 c.c. of 0·0005 *N* iodine solution in a small stoppered tube; then the excess of iodine is titrated with 0·0005 *N* sodium thiosulphate solution.

TESTS ON THE FOREGOING METHODS.

Neither the iodine nor bromate titration of arsenic is new, and both are known to be satisfactory for reasonable quantities of arsenic when the reagents are pure; but when large quantities of reagents are employed and the total arsenic to be estimated is of the order of 0·005 mgrm. of As_4O_6 the influence of impurities becomes important.

Using Bang's method, I have been quite unable to obtain concordant results with small quantities of arsenic, and even the blank is not constant.

The reasons for this appear to be:

(1) Boiling with water does not remove all traces of nitrogen acids; the addition of ammonium oxalate is essential.

(2) Alcoholic solution of phenolphthalein itself absorbs varying amounts of iodine.

(3) The amount of reagents added is too large, so that impurities are introduced.

(4) The sodium hydroxide and bicarbonate used to neutralise the distillate almost always contain some iodine-absorbing substances.

Ramberg's method is much superior to that of Bang. Two points in it are all important, *viz.* the complete oxidation of all traces of organic matter and the removal of all nitrous acid. When these are observed good results are obtained, *provided that the amount of arsenic to be estimated is not too small.* The following table shows results obtained by me with this method; 4 grms. of starch were used in each test.

RAMBERG'S METHOD.

Experiment.	As_4O_6 added. Mgrm.	Bromate solution taken. c.c.	As_4O_6 found. Mgrm.
1.	Nil	0·20	—
2.	Nil	0·20	—
3.	Nil	0·23	—
4.	0·50	2·80	0·52
5.	0·30	1·75	0·31
6.	0·20	1·05	0·17
7.	0·10	0·70	0·10
8.	0·05	0·50	0·06
9.	0·02	0·35	0·030
10.	0·01	0·30	0·020
11.	0·005	0·25	0·010
12.	0·002	0·25	0·010

The results are quite satisfactory down to 0·01 mgrm.; for quantities less than this I find it best to use Ramberg's method of destruction and subsequently the Marsh-Berzelius process; better results are thus obtained than by the older method of the Joint Committee, and there is the advantage that the arsenic mirror is actually seen and may be proved to be arsenic by the ordinary recognised procedure.

So far as the preliminary oxidation treatment is concerned there seems no doubt that the moist combustion recommended by the Swedish Commission is preferable to the older method. When there is a considerable mass of carbon to be extracted with acid, as is frequently the case in the Joint Committee's method, it is very doubtful whether all the arsenic is washed out. From experiments I have made it is almost certain that some is retained or adsorbed by the carbon, as is the common experience in the case of small amounts of copper or lead in foodstuffs. Also, if the heating is continued sufficiently long to ensure a clear and colourless liquid, there is risk of loss of arsenic by volatilisation, and if the liquor is coloured by organic matter, the results of the subsequent estimation are liable to serious error. Naturally these difficulties arise only when considerable quantities of organic materials are being treated.

ARSENIC IN URINE.

Many of the results of the examination of urine reported by the Commission were obtained by Bang; hence, in the writer's opinion, they may be slightly too high. The following results have been obtained by a slight modification of Ramberg's method:

To 100 c.c. of the urine were added 10 c.c. of nitric acid, and the liquid was evaporated in a Kjeldahl flask to about 20 c.c., after which 10 c.c. of sulphuric acid were added, the digestion continued, and the nitric acid removed, as in Ramberg's method, 15 c.c. of ammonium oxalate solution being used. The arsenic was determined in the clear colourless nitrite-free liquid by the Marsh-Berzelius process, and the blank with these quantities of reagents was quite constant at 0·0015 mgrm. The mirrors were estimated in some cases by optical comparison with standards, and when they appeared greater than about 0·01 mgrm. by dissolving them in dilute iodine solution and titrating with thiosulphate; this gives quite good results when compared with the ordinary optical method.

$$As + 5I + 4H_2O = H_3AsO_4 + 5HI.$$

There could be no doubt whatever that the mirrors were of arsenic, as in many instances they were heated in air, with the result that characteristic crystals of As_4O_6 were produced.

Experiment No.	Volume excreted in 24 hours. c.c.	As_4O_6 found. Mgrms. per litre.	As_4O_6 in Mgrms. per day.
13.	1500	0·04	0·06
14.	1200	0·05	0·06
15.	1350	0·03	0·04
16.	1700	0·02	0·03
17.	1600	0·00	0·00
F. 18.	1750	0·10	0·18
19.	1000	0·09	0·09
20.	1200	0·05	0·06
21.	900	0·01	0·01
22.	620	0·03	0·02
23.	1100	0·07	0·08
F. 24.	750	0·25	0·19
25.	950	0·01	0·01
26.	1100	0·02	0·02
27.	1200	0·00	0·00
28.	—	0·02	—
29.	—	0·07	—
30.	1700	0·01	0·02
F. 31.	1050	0·58	0·61
F. 32.	—	0·33	—

For some of these samples I am indebted to Dr. Blackie of the London Homœopathic Hospital.

Samples No. 18, 24, 31, and 32 are from persons having a fish diet; No. 31 had three meals of plaice 24 hours before the sample was taken.

These figures, which are almost as high as those of Bang, show clearly that normal urine may contain quite substantial proportions of arsenic, and confirm the observation that after a fish diet there is an appreciable increase in the output of this metal.

ARSENIC IN FISH.

According to the Swedish Commission the amount of arsenic in fish is sometimes greater than has hitherto been supposed. Bang reports 4 parts per million in one case. In this connection it may be remarked that in the recent Report on Oysters by the Ministry of Agriculture and Fisheries as much as 3·7 parts of As_4O_6 per million was found in certain Whitstable oysters (*cf.* ANALYST, 1924, **49**, 484).

Several kinds of fish have therefore been examined by the Ramberg method of destruction and the arsenic subsequently determined by the Marsh process. The results show that quite substantial amounts of arsenic may be present, although no figures quite as high as those of Bang have been obtained.

It seemed possible that fish feeding in Swedish waters might contain more of this impurity than those from English waters, as sold in the London markets, and to enable me to test this point Prof. Petrén kindly had a case of fish sent from the Swedish Sund. No difference appears between the two sets of figures. The results obtained by the old method of the Joint Committee are set out, side by side with those of the new process and those of Bang and Ramberg, and show that, at least in its application to fish, the old method gives low results. The weight taken for the determinations was 20 to 25 grms. of the edible portion of the fish, including skin but not bone:

ARSENIC AS As_4O_6 PER 100 GRMS. OF FISH.

Fish	Joint Committee's method Mgrm.	Swedish Commission's method (Ramberg) Mgrm.	Amount found by Bang. Mgrm.
(*British*)			
Whiting	0·01	0·04	0·01–0·33
Plaice	0·08	0·14	—
,,	—	0·25	—
,,	—	0·30	—
Sole	0·02	0·03	—
Hake	0·02	0·03	—
Herring	0·01	0·03	0·04–0·08
Cod	0·01	0·05	0·08–0·20
John Dory	0·01	—	—
Haddock	0·01	0·06	—
Brill	0·01	0·03	—
Mackerel	0·02	0·05	—
Halibut	0·00	0·03	—
Turbot	0·02	0·05	0·06–0·18
Perch	—	—	0·08
(*Swedish*)			
Mackerel	0·01	0·01	—
Herring	0·01	0·03	—
Haddock	0·01	0·02	—
Plaice	0·03	0·05	—
,,	0·05	0·09	—
Sole	0·01	0·01	—
Cod	0·02	0·04	—

It is evident that fish may, and often do, contain quite appreciable quantities of arsenic, the amount varying in the different individuals; two fish from the same batch may contain widely different proportions. The high figures obtained from plaice are remarkable; three specimens out of four examined contained more than the Royal Commission's limit of 1/100 grain per lb. one giving twice that quantity. It might be expected that other flat fish, such as the sole, would contain like amounts, but this is not borne out by the results. The reason for this probably lies in the difference in the food of these fish, plaice, as I was informed by Dr. J. H. Orton, of the Marine Biological Association, feeding largely on bivalves, which have been found to contain notable quantities of arsenic; sole feed only to a small extent on bivalves, but largely on worms, starfish and crustacea.

There appears to be ample support for the suggestion that arsenic in human urine is largely due to fish; vegetables and meat have not been found to contain substantial amounts. There is also the proved fact that fish diet gives rise to a large increase in the excretion of arsenic. L. Brahme (*Arsen in Blut*, Stockholm, 1923) claims to have demonstrated arsenic in the blood within an hour of the eating of fish. The source of the arsenic in fish is doubtless due to the food; as has already been noted, plaice get it from bivalves; other kinds get it from other foods or from algæ. In this connection it may be noted that Gautier (*Bull. Soc. Chim.*, 1903, **29** [iii.], 780) and Tassilly and Leroide (*ibid.*, 1911, **9** [iv.], 63) show that algæ, and even sea-water itself, contain this element in recognisable proportions, varying from mere traces up to 0·7 mgrm. in algæ, and 0·08 mgrm. per kilo in sea water.

SUMMARY.—The methods and results of the Swedish Commission on Chronic Arsenical Poisoning have been carefully tested, and the conclusion has been reached that the nitric and sulphuric acid digestion process, described by Bang and Ramberg, gives higher and more accurate results than the older wet combustion method adopted by the Joint Committee of the Society of Public Analysts and the Society of Chemical Industry in 1902. The iodine titration method of Bang is not reliable, but the Ramberg bromate method gives satisfactory results if the amount of arsenious oxide to be estimated is not less than 0·01 mgrm.; below this amount the Marsh-Berzelius method, following the Ramberg method of destruction (details of which are given) is preferable.

It has been found that normal urine may contain quantities of arsenic which have been thought to be associated only with chronic arsenical poisoning; amounts present in the urine of persons on known diets varied from 0·0 up to 0·58 mgrm. per litre. Large excretion of arsenic is due to the eating of fish, and it is shown that fish, especially plaice, may contain arsenic up to 3 parts per million. The eating of such fish leads to the appearance of quantities of arsenic in the urine within twenty-four hours.

DISCUSSION.

Mr. **JOHN WEBSTER** said that in dealing with such very minute traces of arsenic the titration method—with its controls and blanks—seemed to him very unsatisfactory. He, personally, when employing the Marsh and other well-known methods, had not found, under normal conditions, anything like the amount of arsenic mentioned by the author. In his opinion the number of reagents added in the course of the tests was unfortunate, as, naturally, the more reagents added, the more care would have to be exercised as to their absolute freedom from arsenic.

Mr. **Edward Hinks** referred to the opinion of the late Mr. Wilkie, expressed at the Nottingham meeting in 1923, that it was quite usual to find easily-detectable quantities of arsenic in normal urine. As arsenic was so very widely distributed, it was obvious that it must be frequently introduced into the system, and so, presumably, must be excreted in the urine.

Mr. N. T. **Foley** said that, according to his recollection of the Swedish original, Ramberg did check his experiments by the Marsh test.

Mr. J. **Myers** said that it was rather a curious coincidence that the Swedish method described by Dr. Cox was, in its essentials, very similar to the one he was in the habit of employing for the determination of arsenic in pyrites, which method, moreover, came to this country from Sweden, by way of a private communication to Mr. Rudd Thompson. With the reservation that his work on pyrites comprised the determination of percentages of arsenic ranging from 0·004 to 0·5 per cent., and that iodine and starch were employed for titrations, the method was the same, but he would not care to trust it for the extraordinarily minute quantities dealt with by Dr. Cox, because, no matter how careful one might be in the choice of the reagents employed, the blank in his (the speaker's hands) invariably exceeded the quantities of arsenic dealt with in Dr. Cox's paper.

Mr. A. E. **Parkes** asked whether it had been proved that the glass of the apparatus was free from arsenic.

Mr. C. A. **Mitchell** asked whether there was any evidence of the cumulative action of arsenic introduced into the system in fish. It would also be interesting to know in what manner it would be distributed in different organs of the body. It had been proved by Strassmann and Kirstein that, if arsenic were introduced *post mortem* into the stomach, it would accumulate in the left kidney, whilst the right kidney would only absorb traces, so that it would appear that certain organs had a selective action.

Mr. G. R. **Thompson** expressed his personal appreciation of Dr. Cox's work on these Swedish methods, which had been carried out with that scrupulous attention to accuracy with which the speaker had been familiar for so many years when he and Dr. Cox were associated. Apparently nothing was without arsenic content, but he would hesitate to accept the results obtained by a method in which the quantity of reagents added was so large as to introduce the possibility of serious errors.

Dr. **Cox**, replying, said there was no doubt whatever that the substance determined was arsenic, as it had been proved by the formation of arsenic mirrors. With regard to the microchemical method, he, personally, did not endorse what the Swedish Commission had said upon it, and had merely reported their opinion. He had been asked whether the urine of fish eaters might not give a direct result in the Marsh test; it did, and in certain cases the results were checked that way. It was easy to prove that a considerable quantity was present. The method he recommended was the Swedish method of destruction of organic matter by oxidation followed by the Marsh process, so that the effect of the possibility of losses of arsenic—due to the use of the "wet" method only—was eliminated; also it was well known that in tin and lead determinations it was impossible to extract all the tin from a mass of carbon, and in the wet combustion process arsenic was also retained or adsorbed by the carbon. He quite agreed with all that had been said on the quantities of reagents required, but nitric, hydrochloric, and sulphuric acids were all obtainable free from arsenic. Regarding the possibility of there being arsenic in the glass he had used for his experiments, it was possible nowadays to obtain, and he had obtained, glass free from arsenic. As to the cumulative effects of arsenic, the Swedish chemists had determined the amount of arsenic in the fish and the amount of arsenic eliminated by people having eaten the fish, and had found that the amounts agreed; further, they had proved that there was a cumulative effect until the fish diet was discontinued. Mr. Myers had said the methods were not new; that was so; they were only new as applied to foodstuffs, in which the amount to be determined was very much lower than in the case of pyrites.

An Apparatus for the Catalytic Dehydrogenation of Alcohols.

By S. G. WILLIMOTT, Ph.D., B.Sc.

(*Read at the Meeting, November 5, 1924.*)

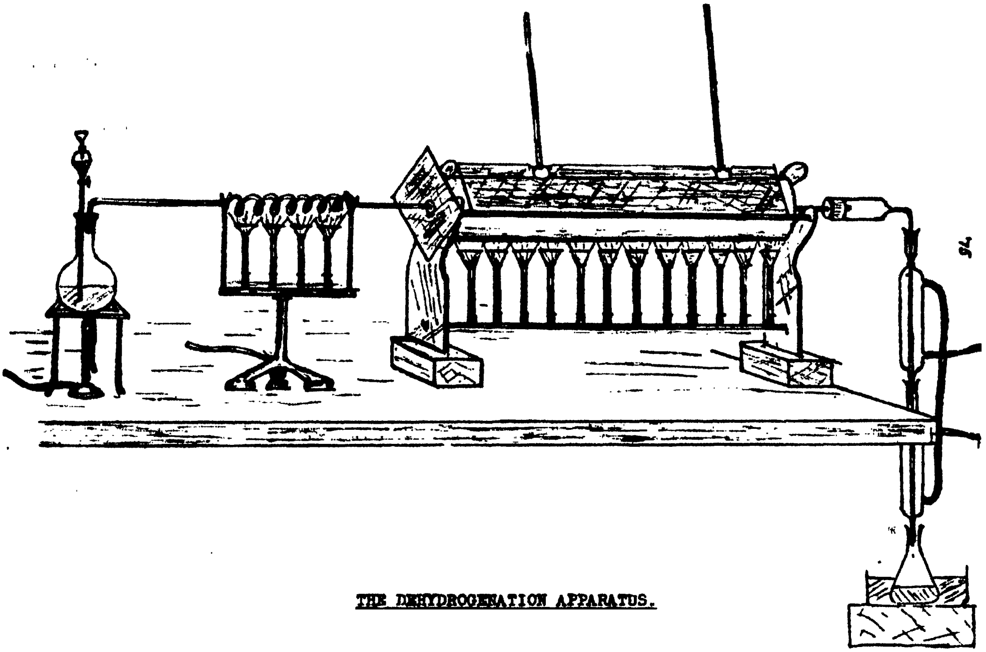
In the course of an investigation upon the synthesis of certain quinoline derivatives, it became necessary to prepare propyl aldehyde in quantity. The usual laboratory methods for the preparation of aliphatic aldehydes by oxidation of the alcohol are unsatisfactory, as the yields are erratic and low. In the present work with propyl aldehyde, for example, I have found that the yield of aldehyde-ammonia by the well-known Gattermann method was, at best, never more than 20 per cent. of the theoretical amount. Similar results have been obtained by Adams and Williams (*J. Amer. Chem. Soc.*, 1921, **43**, 2420), who studied the yields of acetaldehyde obtainable by the Gattermann method. These authors found the yield of aldehyde-ammonia to be only 23 per cent. of the theoretical quantity, and often to be lower than this. They cite Noyes (*Organic Chemistry for the Laboratory*, 1911, p. 89), who predicts a yield of only 15 per cent. for the method. My attention was therefore turned to the catalytic process of dehydrogenation for the preparation of aldehydes and ketones. Neave (ANALYST, 1909, **34**, 346) describes one form of apparatus for carrying out the dehydrogenation of alcohols, as suggested, in the first instance, by Sabatier and Senderens (*Bull. Soc. Chim.*, 1905 [iii.], **33**, 263). A primary alcohol such as propyl alcohol, which was used throughout this work is decomposed in the following way:

$$CH_3.CH_2.CH_2.OH \longrightarrow CH_3.CH_2.CHO + 2H.$$

Neave's apparatus for carrying out a reaction of this type consists essentially of a 28 in. combustion tube of ½ in. diameter, containing a 24 in. layer of finely divided copper prepared from powdered cupric oxide by reduction in a current of dry hydrogen at 300° C. To one end of the combustion tube is attached a bent capillary the vertical portion of which is joined on to a wider vessel. The other end of the combustion tube is connected with a receiver which is cooled by a mixture of ice and salt. An air bath consisting of an iron cylinder 26 in. long serves to regulate the temperature. The bath, which carries two thermometers 12 inches apart, is heated by three Ramsay burners. When the temperature of the tube has reached 300° C., the alcohol (3 c.c.) is poured into the long receiver, from which it should drip steadily into the hot tube, become vaporised and, in its passage through the combustion tube, finally be catalytically decomposed by the copper.

In practice, however, this arrangement for the steady supply of alcohol into the tube was found to be only partly successful, and an improved method was devised. This consisted in driving the propyl alcohol vapour (b. pt. 97° C.) into a well heated copper spiral before its entry into the dehydrogenation tube. This apparatus consisted of a small round flask of 500 c.c. capacity, in which the alcohol was gently boiled, and had a cork with two holes, one for a small dropping funnel, the other for a delivery tube bent at right angles. This was connected by a short piece of rubber tubing with a copper tube of a diameter of ⅛ in. At the end near the combustion tube and remote from the flask, a compact 6 in. spiral was connected directly with the combustion tube, and was heated by a Ramsay burner. An asbestos board protected the cork of the tube from carbonisation by heating of the spiral. The idea underlying this apparatus was simply to secure the alcohol passing over the catalyst as vapour at 300° C.; moreover, with this arrangement no liquid entered the combustion tube, and cracking, which was one of the disadvantages of the Neave apparatus, was avoided. A combustion tube of 40 in. length is preferable to the shorter one used by Neave, as then approximately 36 in. of the catalyst are available for acting upon the alcohol. A cylindrical air bath, 30 in. long and with a diameter of 4 in., was employed to regulate the temperature, and this had two thermometers, 12 in. apart, held in position by well-fitting corks protected by asbestos. A modification was introduced for the heating of the air bath. It was found satisfactory and convenient in practice, to use an ordinary combustion furnace in place of three Ramsay burners for the heating of the air bath. Usually it is only necessary to widen the jaws of the combustion furnace slightly to render it suitable for this purpose. The heating of the air bath and the regulation of its temperature is facilitated by placing a long piece of asbestos on either side of the air bath, as shown in the diagram. With this arrangement, once the required temperature has been reached, a small flame serves to maintain it constant. It is important to have an efficient condensing system for collecting the aldehyde or ketone produced, as otherwise the yield is seriously affected. Two Davis double-surface condensers were found to be efficient, and the receiver itself was allowed to stand in ice-water. The end of the combustion tube is connected with the condensers by means of an adapter, 1 inch in diameter and 8 inches in length, and at this point it narrows to a small tube bent at right angles to connect with the first condenser.

Pure, dry hydrogen, prepared by dropping 10 per cent. alkali on aluminium foil and passing the gas through permanganate solution and sulphuric acid, was used for the reduction of the cupric oxide, which was dry and powdery. The reduction was carried out at 300° C. and the tube kept full of hydrogen until required. The supply to the hard glass tube of the alcohol to be dehydrogenated was easily controlled by simply regulating the boiling. The dehydrogenation was started at 290° C., and the temperature allowed to reach 300° C. as the reaction proceeded. Occasionally it was also found convenient to employ "kupfer-bronz" (Kahlbaum's) when a ready catalyst was desirable. This is especially the case when only a small quantity of material is to be worked upon as, for example, in characterising primary, secondary and tertiary alcohols, as suggested by Neave. The "kupfer-bronz" was applied by intimately coating the walls of the hard glass tube with a uniform layer of the catalyst. The copper alloy, which was in a remarkably fine state of division, readily adhered to the inner surface. The catalytic power of "kupfer-bronz" falls off sooner than when finely divided copper is used, but the yield is not greatly affected. The advantage of this modification is the saving of time, as no reduction is required, and immediately the efficiency of the catalyst becomes impaired it can be easily replaced.



THE DEHYDROGENATION APPARATUS.

The propyl aldehyde was obtained in a pure state by subjecting the liquid in the receiver to two fractionations using a Young column. On redistillation, the aldehyde boiled at 49·5° C. The alcohol remainder was neutralised with calcium hydroxide, as the presence of propionic acid rapidly destroys the activity of the catalyst. It was then redistilled and slowly passed over the catalyst, the process being continued until all the alcohol had been transformed. The average yield was 33 per cent. The complete apparatus is shown in the diagram.

SUMMARY.—1. The usual laboratory methods for the preparation of aliphatic aldehydes are shewn to be unsatisfactory.

2. An improved dehydrogenation apparatus is described, the chief advantages of which are:

(*a*) The alcohol vapour, by passing through a heated copper spiral, enters the tube at 300° C., when it is readily dehydrogenated by the copper catalyst.

(*b*) The spiral pre-heater prevents liquid from entering the tube, with consequent cracking—a disadvantage of the Neave apparatus.

(*c*) The quantity of propyl alcohol vapour entering the dehydrogenation tube is controlled by simply regulating the boiling.

(*d*) The heating of the air-bath is facilitated by employing an ordinary combustion furnace with the introduction of asbestos sheeting which maintains the temperature uniform and reduces the gas consumption.

3. "Kupfer-bronz" can be used as catalyst in small operations.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE COMPOSITION OF COCONUT OIL.

THE publication of remarks by Mr. G. D. Elsdon (ANALYST, 1924, **49**, 423) on the determination of the composition of the mixed fatty acids of fats by the method of fractional distillation of the methyl or ethyl esters has brought to light an omission from the table contained in the Presidential address to the Society of Chemical Industry, recently delivered by the retiring President, Dr. E. F. Armstrong (*J. Soc. Chem. Ind.*, 1924, **43**, 207T), which shows the percentage composition of fatty acids derived from certain fats.

Owing to the unfortunate omission of an identifying numeral the composition of coconut oil therein quoted, which is the work of E. Paulmyer (*J. Soc. Chem. Ind.*, 1907, **26**, 881) has not been attributed to him. This is the more unfortunate, as Mr. Elsdon quotes these figures in his comments.

As was stated in the text of the address, the authors are in agreement, to a considerable extent, with Elsdon and with Channon, Drummond and Golding, that the method is capable of giving only an approximation to the true composition of the fat under examination, but they are convinced that many of the obviously erroneous results are to be attributed to the following causes:

(1) The use of more or less imperfect fractionating apparatus, and an endeavour to arrive at fractional separation of the mixed esters which

have been prepared from fatty acids before any attempt has been made to separate the saturated and unsaturated constituents of the original fatty acid mixture.

(2) Assumptions as to the composition of the fractions obtained without having identified the fatty acids which are assumed to be present in them.

It is proposed to publish in the near future the results of an extended investigation, now nearly completed, of the fatty acids obtained from coconut oil, in the carrying out of which special precautions have been taken to avoid, as far as possible, the sources of error which surround this particular method of analysis.

JOHN ALLAN.

DETERMINATION OF MILK SUGAR IN MILK.

THE following details for carrying out the iodimetric determination of milk sugar in milk (*cf.* Kolthoff, ANALYST, 1923, 48, 386) have been found to work well.

Ten c.c. of milk are weighed into a 100 c.c. flask and diluted with 50 c.c. of distilled water; 10 c.c. of Mayer's reagent and 2 c.c. of *N* sulphuric acid are added, the whole well shaken and the volume made up to 100 c.c. The solution is filtered, and 25 c.c. of the filtrate are neutralised to phenolphthalein (1 drop used); then 20 c.c. of 0·1 *N* iodine solution and 30 c.c. of 0·1 *N* sodium hydroxide solution are added. After 20 minutes, 4 c.c. of 0·1 *N* sulphuric acid are added, and the residual iodine titrated with 0·1 *N* thiosulphate solution.

The percentage of anhydrous milk sugar by weight is calculated thus:

$$\text{Per cent.} = \text{c.c. iodine used} \times 0{\cdot}0682 \times \frac{100-(0{\cdot}3+\text{Fat}\times 1{\cdot}11)}{\text{Weight of milk}}.$$

If desired, 10 c.c. of milk may be measured, and the weight of this taken as 10×sp. gr.

For the determination of milk sugar in condensed milk 10 c.c. of a mixture of 1 part of condensed milk and 2 parts of water, both by weight, are used, or about 3·5 grms. are weighed direct into the flask.

H. DROOP RICHMOND.
L. R. ELLISON.

THE DETERMINATION OF FAT IN CONDENSED MILKS.

FOR the determination of fat in condensed milks the Röse-Gottlieb method is usually recommended. The Werner-Schmidt method gives low and variable results, especially in the case of sweetened condensed milk, with which there is a considerable amount of charring of the sucrose.

The following modification of the latter process was devised to overcome this difficulty, and has given very satisfactory results. It depends on the fact, embodied in Ritthausen's process, that the sugars can be filtered off after addition of copper sulphate solution to an emulsion of the milk, the fat remaining entangled in the precipitate. Copper sulphate was chosen as being the most convenient solution to use, but any other protein precipitant would probably serve equally well.

From 2 to 3 grms. of the condensed milk are weighed into a fat flask and completely emulsified by means of a glass rod with about 20 c.c. of cold water. Five c.c. of Fehling's copper sulphate solution are added, and the flask placed on the water bath for a few minutes to complete the precipitation of the proteins. The contents are then filtered through a wet ribbed filter paper, the flask being washed out with warm water and the washings passed through the filter paper. Some of the precipitate remains adhering to the walls of the flask and need not be removed. The filter paper containing the precipitate is transferred to the flask, and 10 c.c. of hydrochloric acid are added. The filter paper is roughly broken up with the glass rod, and the rod, after washing, is then removed. The flask is placed on the water bath until the fat can be seen as clear globules on the surface of the liquid. Half an hour is usually sufficient. The filter paper by now is completely disintegrated and no charring takes place. The fat is obtained by three extractions with petroleum spirit, the extract being washed to remove traces of acid. The petroleum spirit is evaporated, and the fat weighed after the flask has been in the oven for about an hour.

The results obtained by this method have been checked by a second determination by means of the Röse-Gottlieb process, and in each case the two results have been in close agreement.

R. W. Sutton.

A CASE OF PRUSSIC ACID POISONING WITH LINSEED CAKE MEAL.

A sample of linseed cake meal which was alleged to have caused the death of a calf nine weeks old, and to have produced "stomach staggers" in the case of another calf, was recently sent in for examination. The food had been given in the form of a gruel made by soaking the meal for 12 hours in cold water.

The "free" prussic acid was determined by the method given in Auld and Ker's *Practical Agricultural Chemistry*, and 0·054 per cent. was found. This quantity is exceedingly high—in fact, the highest that has come to the writer's notice. There is no doubt that the method of preparation of the gruel, coupled with a cake of high glucoside content and enzymic activity, was responsible for the death of the animal.

H. T. Cranfield.

The Midland Agricultural and Dairy College,
Sutton Bonington, Loughborough.

DETERMINATION OF CADMIUM IN SPELTER AND ALLOYS.

In view of the need of an expeditious method of determining cadmium in spelter, etc., it might be worth while to investigate the applicability of the precipitation of tetramminecadmium iodide (Analyst, 1917, **42**, 189) to the quantitative separation of cadmium from zinc. The scheme I have in mind consists in precipitating cadmium sulphide, mixed with a certain amount of zinc sulphide, from an ammoniacal solution by cautious addition of dilute sodium sulphide solution. The precipitate is dissolved in hydrochloric acid, the solution is evaporated, and the dry residue is taken up in a few c.c. of tartaric and dilute nitric acid solution. The cold solution is then treated with 30 c.c. of strong ammonia and 4 grms. of solid potassium iodide (Analyst, 1919, **44**, 275). The white crystalline precipitate is filtered off and washed free from zinc with a 4 per cent. solution of potassium iodide in ammonia (4:1 water); it is dissolved in a little sulphuric acid, and the cadmium determined as phosphate (Analyst, 1916, **41**, 124). The proposed method follows the lines of the separation and estimation of cobalt, described in the papers referred to above.

W. R. Schoeller.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

LABELLING OF MARGARINE.

On November 7 the Glasgow Stipendiary Magistrate heard the arguments of counsel in a case brought before him under the Butter and Margarine Act, 1907, The facts were not in dispute, and the sample was proved to be margarine, but the point to be decided was whether the words on the wrapper, "Containing a small quantity of butter, Dutch made," formed part of the descriptive name of the article. These words were in small type beneath the words "Charmo Margarine" in large type, the name "Charmo" being a fancy name approved by the Board of Agriculture and Fisheries, and permitted to be combined with the word "Margarine" under Sec. 8 of the Butter and Margarine Act, 1907.

In giving his decision, the Stipendiary said that he had come to the conclusion that the words in question were part of the descriptive name of the accused's margarine, and that they were a contravention of Section 8 of the Butter and Margarine Act, 1907, as such name had not been, and admittedly could not be, approved by the Board of Agriculture and Fisheries. Counsel for the accused had submitted that there was a distinction between this case and the case of *Maypole Dairy Company* v. *Paterson* (1923, J.C. 85), and that this case was on all fours with the case of *Hawes* v. *Stephens* (K.B.D., May, 1924). He (the Stipendiary) could not see any difference between this case and that of the Maypole Dairy Company. The words in that case were "Mixed with Maypole butter," and he could see no difference between those words and the words in this case, *viz.* "Containing a small quantity of butter, Dutch made," which, in his opinion, were so associated with the words "Charmo Margarine" as together to constitute a descriptive name of the accused's margarine in the sense of the section libelled.

Accordingly he imposed a fine of £5, but agreed to state a case for appeal.

CUSTARD WITHOUT EGG.

On November 5th a grocer was summoned at Durham County Petty Sessions for the sale of custard that was not of the nature, substance and quality demanded.

The Deputy Clerk of the Peace stated that six penny packets of "custard" had been purchased from the defendant, and that these, on analysis, had been found not to contain a mixture of eggs and milk. Although no standard had been laid down for custard, as in the case of milk, reference to the dictionaries showed that "custard" was "a mixture of eggs and milk."

The County Analyst (Mr. C. J. H. Stock) gave evidence to the effect that the samples analysed by him consisted entirely of maize starch, coloured and flavoured.

Dr. Hill, Medical Officer to the County Council, said that, in his opinion, the basis of all custard was egg, and that, in the making of custard, milk was a necessity.

The solicitor for the defence pointed out that not one person in a thousand went into a shop and asked for a box of custard. In this case the word "powder" had been accidentally omitted from some of the smaller labels. There were on the market any number of powders which did not contain egg or milk. These ingredients were added afterwards.

The Court convicted the defendant, and imposed a fine of £3.

MILK "AS IT CAME FROM THE COW."

DAVIS *v.* BLACKMAN.

ON November 11th an appeal was heard in the Divisional Court, before the Lord Chief Justice and Justices Shearman and Salter, from a decision of the Southampton County Justices. The defence had been set up that the sample of milk, which had been found to be deficient in milk fat, was sold "as it came from the cow," and the question raised was whether this plea was sufficient to rebut the prosecution.

Mr. W. Blake Odgers, counsel for the appellant, an inspector under the Sale of Foods and Drugs Act, stated that the guidance of the Court was desired upon the question whether the justices should have allowed analytical evidence to be given to show the composition of other samples of milk produced under the same conditions in the same part of the country. The justices had excluded such evidence on the ground that it was irrelevant.

In giving his judgment the Lord Chief Justice said that in recent years the formula "as it came from the cow" had come to be regarded as almost of sacramental efficiency in cases relating to the adulteration of milk. In this case the appellant had sought to give evidence for the purpose of showing that the justices ought to be exceedingly careful before accepting evidence intended to show that milk was being sold in the condition in which it came from the cow. The reason given by the justices for not admitting this further evidence was that the appellant was confined, by the form of the information, to the regulations of the Board of Agriculture. In his opinion this view was erroneous.

The other Justices concurred, and the case was remitted to the Southampton justices for further consideration.

Queensland.

REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR ENDED JUNE 30, 1924.

THE number of samples examined during the year was 6817, of which 1438 were for the Customs, 1533 for the Department of Health, 1606 for the Postmaster, and 991 for the Mines Department. The drop of 771 in the number of mines samples was mainly due to fewer samples being sent from the State arsenic mine.

Of the 615 legal samples taken by inspectors under the provision of the Health Acts, 367 were passed, 165 failed to reach the standard, and 62 were unfit for analysis. There were 38 prosecutions (30 for milk, 5 for spiritous liquors and 3 for poisons) and convictions were obtained in every case The large proportion of "failures" to reach the standard arose through these samples having been found by the inspectors to be close to or below standard in their preliminary examination.

Of the 525 legal samples of milk, 65·5 per cent. passed the standard, 12·8 per cent. were adulterated with water, 3·6 per cent. were deficient in fat, 11·8 per cent. were unfit for analysis, and 6·3 per cent. were genuine, but slightly below standard.

INFANTS' FOODS.—The following analyses of infants' foods on the local market were made in order to ascertain their position with regard to the standard for infants' food suggested by the 1922 Food Conference.

Food.	Water. Per Cent.	Protein. Per Cent.	Fat. Per Cent.	Carbohydrate. Per Cent.	Mineral Substance. Per Cent.	Cold Water Extract. Per Cent.	Crude Fibre. Per Cent.	Remarks.
Glaxo	3·2	24·1	26·6	40·5	5·6	—	—	Desiccated milk with added lactose.
Lactogen	2·7	25·0	26·2	40·4	5·7	—	—	Desiccated milk with added lactose.
Horlick's Malted milk	5·1	12·5	7·9	70·9	3·6	74·5	Nil	Desiccated milk and desiccated malt extract.
BacchusMarsh malted milk	3·7	11·2	10·8	71·2	3·1	—	—	Desiccated milk and desiccated malt extract.
Mellin's Food	5·6	10·2	0·9	80·6	2·7	82·8	—	Desiccated malt extract.
Benger's Food	5·4	10·9	0·8	81·9	1·0	16·8	Nil	Wheat flour and pancreatic extract; very little unchanged starch when made as directed.
Savory and Moore's	6·7	9·8	1·2	81·5	0·8	—	—	Wheat flour and malt extract; most of starch changed when prepared as directed.
Allenbury's Malted Milk	4·9	9·7	0·8	83·5	1·1	—	—	Wheat flour and malt extract; most of starch changed when prepared as directed.
Nestles' Milk Food	3·3	12·8	4·1	78·1	1·5	66·0	0·2	Desiccated milk, bakedwheat flour, and cane sugar.
Neave's Food	3·2	13·4	1·2	81·1	0·7	6·6	0·4	Baked wheat flour.
Australian Groats	3·3	9·8	7·2	78·4	0·8	—	0·5	Ground oats.
Robinson's Groats	7·6	12·3	6·8	70·4	1·5	—	1·4	Ground oats.

COCOA POWDER.—Cocoa powder for use in a tropical climate should not contain more than 5 per cent. of water, as a proportion in excess of this limit tends to make the powder lumpy and liable to attack by fungoid growths. Several of the samples examined were lumpy and deteriorated owing to the water content being above the safety limit. The samples were free from foreign fat and added starch. Several of them contained fibre considerably in excess of the maximum proportion permitted to be present.

LIQUID PARAFFIN.—Only one sample of liquid paraffin failed to conform to the B.P. standard. This standard, however, is not comprehensive enough, in that it does not take cognisance of what is probably the most important feature of liquid paraffin for internal use, *i.e.* viscosity; a thin oil traverses the system rapidly and it is possible that, by reason of its solvent nature, it would be injurious. The samples examined ranged in viscosity from 62 to 212. The *Lancet* reported figures on commercial samples ranging from 67 to 440 and recommended a viscosity of at least 105 and a gravity not less than 0·88 (B.P. 0·86 to 0·89).

J. B. HENDERSON.

Government of Palestine.

ANNUAL REPORT OF THE DEPARTMENT OF HEALTH FOR THE YEAR 1923.

THE sections in this Report include Vital Statistics and Epidemic Disease, Medical Services, Sanitary Administration, Relief, Quarantine, and Laboratory Work comprising (*a*) Bacteriological, (*b*) Chemical, and (*c*) Work at the Antirabic Institute.

CHEMICAL DIVISION.—A summary of the work done during the year is given by Mr. G. W. Baker, the Government Analyst. Most of the Government Departments made use of the laboratory, and, in all, 4176 samples were examined. Of these, 3497 samples were milk, of which 846 were taken in the Jerusalem district. There were 236 prosecutions for the sale of watered or skimmed milk.

BUTTER.—The articles sold as butter in Palestine may be divided into three classes:

1. Australian butter imported chiefly for those merchants who cater for non-Palestinian customers. The three samples examined conformed to the English standard for butter.
2. Semni (or cooking butter) manufactured locally. This should consist entirely of butter fat from the milk of the cow, goat or sheep, and should be practically free from casein and water. Genuine semni commands a good price, and therefore invites adulteration with cheap fats; and a large proportion now offered for sale is mixed with, or consists entirely of, vegetable fats or oils.
3. Artificial butter. There are numerous butter factories producing a blend of milk with a vegetable oil or fat. These products can be purchased in printed wrappers marked "Artificial butter," "Almond butter," etc., but there is no doubt that they are also sold loose under the name of "butter" alone. Out of ten samples purchased as butter in the Jaffa district, nine proved to be composed of fat other than butter fat and contained an excessive amount of water. Under a new ordinance it will be possible to fix standards and take steps to prevent the sale of spurious articles.

CANNED ARTICLES.—Samples of jam of local manufacture have been found to contain zinc. Unused cans from the factory have been examined and zinc chloride found in the seams inside the can. Zinc chloride had evidently been used as a soldering flux. The manufacturer stated that he would shortly be in a position to use properly made cans. In the meantime he has been instructed to use rosin in the place of zinc chloride.

The importation of pickles from Egypt contained in old petrol tins has been investigated with the assistance of the Public Health Authorities in Cairo. Steps have been taken to prohibit the importation of pickles in metal containers.

ALCOHOLIC LIQUORS.—Further investigations into the trade in spurious liquors have been carried out. It has been established that potable spirits are manufactured in large quantities from "industrial" or refined spirit imported in iron drums at a strength of 96 per cent. alcohol. This alcohol is watered down to about 40 per cent. and suitable flavouring materials are added. It is sometimes distilled, but more often the "cold process" is employed, which means simple dilution without distillation. This diluted alcohol is then bottled and labelled; this is done both at the distillery and at retail establishments. Cognac, arak, rum, and whiskey are all made by this process.

A large variety of spurious labels has been found in the bottling establishments. These are mostly printed in France. Bottles filled from one cask of spirit may be labelled differently and sold at different prices. The statements on the labels are calculated to deceive the purchaser into the belief that he is buying a genuine imported article.

Measures of control have been considered, and, pending their application, prosecutions have been instituted in certain cases under a section of the penal code. These are looked upon as test cases. They are still in the Courts.

DEAD SEA WATER.—Experiments on the evaporation and crystallisation of water from the Dead Sea have been carried out, and a sample of carnalite was prepared from the water to be sent to the British Empire Exhibition.

LEGAL AND CRIMINAL INVESTIGATION.—The following poisons have been detected in cases of human poisoning:—Arsenic (1), mercury (1), alcohol (1), mydriatic alkaloid (seed of *Datura Metel*) (3), hydrastine (1), opium (1), and chloral hydrate (1). Arsenic was found in 13 cases of cattle poisoning.

The seeds of *Datura*, when used as a poison, are generally crushed and inserted into the middle of dried figs; robbery is usually the motive in these cases. Dried figs are also a favourite vehicle for arsenic.

A counterfeit coin contained 26 per cent. of silver, the remainder being chiefly copper and antimony.

The Problem of Atmospheric Pollution.*

IN the year 1923 there were 3262 deaths in Salford, more than a third of which were due to respiratory disease, including lung consumption. Comparison of the borough statistics with those of England and Wales, as a whole, shows that the percentage of deaths from non-tubercular respiratory diseases in Salford is unduly high. The corresponding death rates from this cause per million persons living were:—Salford, 3092; England and Wales, 1867. The charts for years free from influenza epidemics (1916 and 1917) show a close relationship between fog periods and deaths from respiratory diseases, and confirm the results of clinical experience of the effects of smoke fog on patients whose respiratory organs are already damaged.

MEASUREMENT OF ATMOSPHERIC POLLUTION.—This is now systematically carried out by means of the "Standard Gauge," followed by analysis. The results for each month at four stations, as determined by Elsdon, are given in tabular form. The total and average monthly impurities for the 6 months from October, 1923, to March, 1924, were as follows, the figures representing metric tons per sq. kilometre:

	Tar.	Other insoluble carbonaceous matter.	Soluble matter. Loss on ignition.	Ash (soluble and insoluble).	Total solids.
Total, 6 months	1·45	17·07	13·75	47·13	79·40
Average, 1 month	0·24	2·84	2·29	7·86	13·23

Assuming the amount of atmospheric deposit at one of the Salford stations to represent an average for the Borough, the amount of "dirt" deposited was at the rate of about 3000 tons per annum.

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* By H. Osborne, M.D., Medical Officer of Health, Salford.

The estimated consumption of coal in factories and in houses in the Borough is as follows:—Factories, 5877 tons per week all the year round; houses, 7077 tons per week (summer) and 11,305 tons per week (winter). In addition to this, the efficiency of combustion of household fires is only 65 per cent., as compared with over 90 per cent. in factories. Hence it would appear that the domestic fire is a greater cause of pollution than the factory fire.

CHARACTER OF POLLUTION FROM DOMESTIC FIRES.—In recent investigation on the problem of air pollution by domestic fires, carried out for the Manchester Air Pollution Board, the following comparative analyses were made by E. F. Greig:

ANALYSIS OF FLUE GASES from (1) A Coal Fire and (2) A sitting-room Gas Fire, per 1000 B.Th.U. available in fuel, the two fires being at maximum efficiency.

Unburned Gases, &c.	Coal Fire.	Gas Fire.
Carbon dioxide	1·38 cubic feet	1·03 cubic feet
Carbon monoxide	0·04 ,,	0·10 ,,
Methane	0·02 ,,	0·05 ,,
Unsaturated hydrocarbons	0·03 ,,	0·08 ,,
Soot	43·2 grains	2·6 grains

These results bring into prominence the fact that an ordinary gas fire produces much more carbon monoxide than a coal fire. They do not include the sulphur products (which are capable of setting up respiratory diseases) in the flue gases from either fire.

THE REMEDY.—It is not probable that the gas fire or radiator will replace the domestic fire, and from many points of view (*e.g.* health, value of visible rays) it is not advisable that they should do so. The solution of the problem appears to lie in the adoption of special carbonised fuel to be burned in open grates. The effect of carbonisation on the proportion of sulphur is shown in the followng results:

1.	Sulphur contained in raw Lancashire coal	2·93 per cent.
2.	,, ,, Smokeless fuel made from (1) without use of steam	2·14 ,,
3.	,, ,, Smokeless fuel made from (1) with use of steam	1·26 ,,

The steam motor wagon is a frequent cause of pollution, and since the prosecution of an owner, on June 2, 1920, it has been almost impossible to get a conviction. In that case, it was successfully contended that no penalty could be imposed, as the emission of smoke was due to "temporary or accidental causes," as stated in Locomotive on Highways Act, 1896. To be successful in a prosecution it is now necessary for an inspector to follow the steam wagon, and to prove that smoke was repeatedly emitted during its passage through the Borough.

Ministry of Health.

CIRCULAR 553. SALE OF FOOD AND DRUGS ACTS, &c.

THE following circular has been sent to the Clerks of Authorities administering the Food and Drugs Acts:—

SIR,

1. I am directed by the Minister of Health to request that a copy of the Report of the Public Analyst for the last quarter of the present year and a copy of the Medical Officer of Health's Report upon the administration of the Public Health (Milk and Cream) Regulations during the year, may be forwarded during the month of January.

2. An instance has been brought to the notice of the Department in which a part of a sample of dried milk taken under Article 5 of the Public Health (Dried Milk) Regulations, 1923, was placed in a paper envelope. The consequence was that, by reason of the absorption of water from the atmosphere into the dried milk and of fat from the dried milk into the envelope, the result of the analysis would not represent the actual composition of the article at the time of sale. It is desirable that samples of articles of this character should be placed in securely sealed bottles or other non-absorbent and air-tight containers, so that there may be no material change in the composition of the sample between the time of purchase and the time of analysis.—I am, etc.,

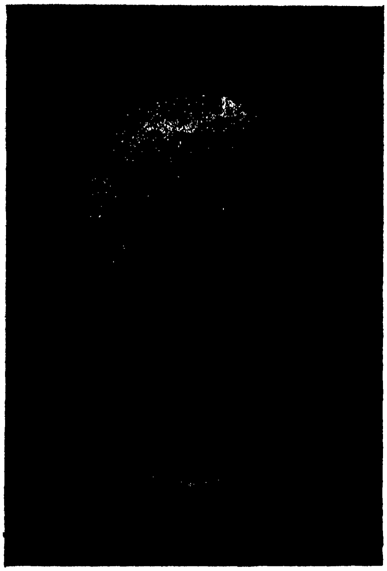
R. B. CROSS, *Assistant Secretary*.

December 29, 1924.

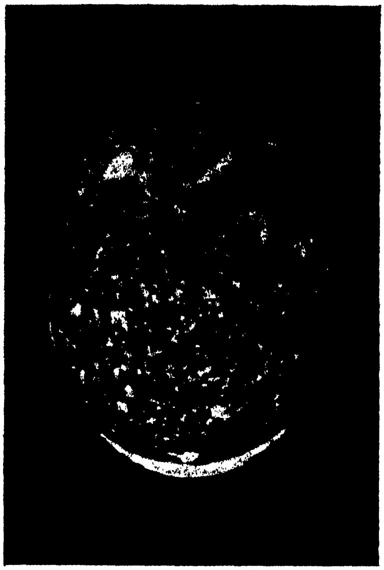
ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Modifications in the Transparency of the Shells of Eggs on Keeping. A. M. Leroy. (*Ann. Falsificat.*, 1924, **17**, 407–408.)—When a newly-laid hen's egg is examined by strong transmitted light (*e.g.* from a 50 c.p. metal filament lamp), the shell shows a uniform appearance, with darker points here and there due to calcareous granulations, the number and distribution of which are fairly constant for all the eggs from the same hen; about 10 to 12 hours after laying, light patches begin to appear on the dark background of the shell, and by the following day these show well-defined margins, and these are generally more concentrated towards the poles of the egg than in the intermediate zones. They are still more pronounced on the next day, but from that time onwards keep the same appearance until obvious alterations appear. By this means it is possible to distinguish between eggs laid the same day and those laid two or three days previously. There are pronounced variations in the number, relative size, and distribution of the light patches in the shells of the eggs of different hens, but these characteristics are remarkably similar in the case of eggs from the same hen. The patches are independent of the protein membranes in the egg, and experiments have indicated that their formation is due to certain parts of the shell being hygroscopic, whilst other parts rapidly lose their water of constitution by evaporation and become darker in colour. Their appearance is promoted by heat and retarded by humidity. Eggs kept in an atmosphere saturated with moisture will keep their transparency indefinitely.



Newly laid Egg.



Egg, 12 hours after laying.

Colorimetric Determination of Sucrose in the presence of other Sugars. H. Riffart and C. Pyriki. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 197–207.) —After a detailed discussion on the influence of temperature, time, and concentration on the colour produced by various sugars with sulphuric acid, it is shown that sucrose can be determined with an accuracy of about ±0·5 per cent. in the presence of other sugars by means of the colour produced by 70 per cent. sulphuric acid at 55° C. The colour is really due to fructose, and so is given by those sugars that produce fructose under the conditions of experiment; the method is therefore available in the presence of glucose, lactose, maltose, or galactose. Albumin or fat must first be removed, and when this is done good results are obtained in the determination of sucrose in condensed milk, biscuits, cake, chocolate, or beer. From 5 to 20 grms. of the material are made into a cream with water, treated with a clarifying agent diluted to 100 c.c. and filtered; 5 to 10 c.c. of the clear colourless filtrate are mixed with twice their volume of 70 per cent. sulphuric acid and heated on a water bath at 55° C. ± 1° for exactly 10 minutes, the thermometer being placed in the liquid as soon as that temperature is reached; the mixture is then cooled and the colour compared in Nessler glasses with that of a standard sucrose solution similarly treated. The more strongly coloured solution is diluted as required, but if its volume exceeds 50 c.c. it is advisable to start again with a fresh and smaller quantity. In the case of milks or chocolate the casein is removed with an acid mercuric chloride solution in the usual way. H. E. C.

Direct Method for the Determination of Glucose and other Carbohydrates. E. Knecht and E. Hibbert. (*J. Chem. Soc.*, 1924, 125, 2009–2012.)—By increasing the molecular proportion of phenylhydrazine to sugar to 12:1 the osazone is quantitatively formed and may be titrated with accuracy by means of titanous chloride without actually being isolated. To 10 c.c. of a 0·1 to 0·2 per cent. solution of the sugar are added 1 c.c. of saturated sodium tartrate solution and 0·25 grm. of phenylhydrazine dissolved in acetic acid; the mixture is heated for ten minutes in a briskly boiling water bath, then a large excess of standard titanous chloride solution is added, and the solution boiled over a free flame for two minutes, a current of carbon dioxide being passed into the flask meanwhile. The excess of titanous chloride is then titrated hot with a solution of crystal violet until a permanent red colour is produced. The crystal violet solution is standardised against the titanous chloride sugar, the equivalent of which can be calculated from the equation:—

$$C_{18}H_{22}O_4N_4 + 6H + H_2O \longrightarrow C_6H_{13}O_5N + NH_3 + 2C_6H_5NH_2.$$

Lævulose and invert or other sugars forming osazones may be similarly determined. H. E. C.

Acidity of the Oil in Olives. R. Marcille. (*Ann. Falsificat.*, 1924, 17, 398–400.)—The pulp of about 20 olives is crushed in a mortar, with the addition, from time to time, of small quantities of neutral anhydrous sodium sulphate until the mass is no longer sticky. Then, after standing for 2 or 3 minutes, the paste is covered with anhydrous redistilled carbon disulphide, and the grinding repeated. The extract is filtered, the residue washed two or three times with the solvent, so as to obtain in all about 50 c.c. of filtrate and washings. These are collected in a weighed glass basin, allowed to evaporate spontaneously; the residue is weighed and redissolved in neutral ether, and the solution titrated with standard alcoholic sodium hydroxide solution (phenolphthalein as indicator); or the residue may be dissolved in a neutral mixture of benzene and 95 per cent. alcohol (2:1), and Poirrier blue C4B used as indicator. Carbon disulphide is the best solvent, because it is not easy to remove petroleum spirit completely from the residue, and benzene and ether extract constituents other than oil from the epidermis of the fruit. For example, the following amounts of acid (in terms of oleic acid) were determined in the material extracted with these various solvents from the same batch of olives: Carbon disulphide (spnotaneous evaporation), 0·39; carbon disulphide (final evaporation in oven), 0·36; petroleum spirit, 0·36; benzene, 1·56; and ether, 2·18 per cent. The acidity of the oil in fresh olives of other varieties cultivated in Tunis ranged from 0·29 to 0·50 per cent. Olives delivered to the works for expression contained oil with an acidity of 0·62 per cent., rising to 1·3 per cent. after 6 days, and 2·18 per cent. when the fruit was bruised. The increase in the acidity was due to the lipolytic action of the mould, *Penicillium glaucum.*

Pectin in Fruits and Fruit Products. H. J. Wichmann. (*J. Ass. Off. Agric. Chem.*, **1924, 8,** 123–130.)—The material is pulped by passing it through a meat grinder, care being taken neither to crush seeds nor to lose liquid. After thorough mixing, 300 grms. are boiled with 800 c.c. of water for 1 hour in a 1·3 to 2 litre beaker, the evaporated water being replaced occasionally. The mass is then transferred to a 2000 c.c. measuring flask, cooled, made up to volume, and filtered through a folded paper. To 200 c.c. of the filtrate are added 2 to 4 lumps of cube sugar if the solution does not contain sugar; 25 c.c. are evaporated and treated with 200 c.c. of 95 per cent. alcohol, allowed to settle and filtered, and the precipitate washed with 95 per cent. alcohol and transferred to the original beaker with hot water. The pectin solution is evaporated to 40 c.c., cooled to 25° C. or below, and, if water-insoluble substances separate during evaporation, it is stirred vigorously and, if necessary, treated with a few drops of 10 per cent. hydrochloric acid and warmed. It is cooled again, and from 2 to 5 c.c. of 10 per cent. sodium hydroxide solution, mixed with sufficient water to make a total volume of 50 c.c. are added, the bulk of the precipitate indicating approximately the quantity to use. After the lapse of 15 minutes, 40 c.c. of water and 10 c.c. of 10 per cent., hydrochloric acid, are added, the liquid boiled for 5 minutes, and the pectic acid filtered off on a qualitative filter-paper and washed with hot water. Filtration should be rapid and should yield a clear filtrate; if it is cloudy or colloidal, the determination should be repeated with addition of more alkali. The pectic acid is washed back into the beaker, the contents of which are adjusted to 40 c.c., cooled to below 25° C., and the saponification and precipitation repeated as described. The liquid is filtered and the pectic acid washed with hot water only until the filtrate shows a negligible amount of acid; more than 500 c.c. of total filtrate should be unnecessary. Finally the pectic acid is washed into a platinum dish and dried on a steam-bath and afterwards in a water-oven to constant weight. It is ignited, and weighed again; the loss in weight represents pectic acid.

T. H. P.

Optical Rotations produced by Lemon Oil and Sweet Orange Oil in Alcoholic Solution. W. W. Randall. (*J. Ass. Off. Agric. Chem.*, **1924, 8,** 206–214.)—To find the approximate percentage of lemon oil by volume in an alcoholic extract of lemon, the angular degrees of rotation of the extract at 20° C. in a 200 mm. tube should be divided by 1·12, or the Ventzke degrees by 3·22; for sweet orange extract the corresponding divisors are 1·81 and 5·21 respectively.

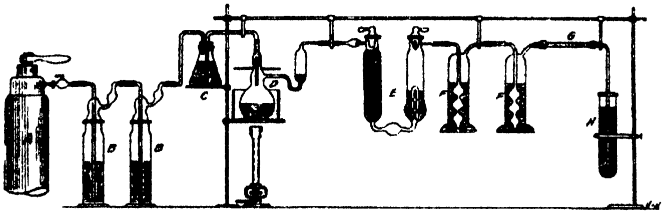
T. H. P.

Acids of Whortleberries and Tamarinds. H. Kaiser. (*Chem. Zeit.*, 1924, **48**, 700–701.)—The acids of whortleberries and tamarinds have been investigated and, on the assumption that the losses by the individual acids at the completion of the work are always proportionately equal, they were present in the following amounts:—

Whortleberries.	Per Cent.	Tamarinds.	Per Cent.
Lactic acid	0·81	Lactic acid	0·007
Oxalic acid	0·16	Oxalic acid	0·022
Succinic acid	4·87	Succinic acid	0·16
Malic acid	18·70	Malic acid	2·34
Citronic acid	72·38	Tartaric acid	96·68
Quinic acid	2·68(?)	Citronic acid	0·04
Unsaturated acids ..	0·40(?)	Unsaturated acids ..	0·75(?)

The method of separation used was based on the difference in solubility of the hydrazides and benzylidene compounds of the respective acids. These esters were prepared after defecation of the juice with lead acetate and separation of pectins by means of alcohol. P. H. P.

Volatilisation Method for the Determination of Fluorine in Baking Powder. J. K. Morton. (*J. Ass. Off. Agric. Chem.*, 1924, **8**, 101–105.)—Twenty grms. of the powder are heated in an 11 cm. porcelain dish in a muffle furnace at a temperature causing no discernible reddening (about 450° C.). The dark ash is powdered, evaporated with 5 c.c. of 10 per cent. magnesium nitrate solution and again heated as before; this operation is repeated once, and the ash then kept in a desiccator until used. The further procedure, which is a modification of that given by Wagner and Ross (*J. Ind. Eng. Chem.*, 1917, **9**, 1116), involves the use of an apparatus comprising a source of air, carbon dioxide, or hydrogen at constant pressure; a gas-drying apparatus; a flask of about 100 c.c. capacity, from which the anhydrous sulphuric acid may be forced by the gas into the digestion flask, which consists of a 4 oz. pyrex distilling flask with a small trap and reflux tube at its outlet; a Schmidt's tube containing glass beads in one arm and, in the other, a 10 per cent. solution of silver sulphate in anhydrous sulphuric acid;



APPARATUS SET-UP FOR THE DETERMINATION OF FLUORINE IN BAKING POWDER.

Bowen absorption bulbs containing a saturated solution of dry chromic acid in anhydrous sulphuric acid; a horizontal glass tube, 6 by $\frac{3}{8}$ inches, containing glass wool; a test-tube containing water, into which the silicon fluoride is delivered. The ash, together with 1 grm. of quartz flour, previously digested with concentrated hydrochloric acid, washed free from acid, and dried, and 5 grms. of anhydrous copper sulphate, are introduced into the digestion flask, and mixed thoroughly, the flask being replaced. After 75 c.c. of anhydrous sulphuric acid have been placed in the 100 c.c. flask and 15 c.c. of water in the final test-tube, the gas is turned on very slowly and the pressure allowed to force the acid into the digestion flask; the first portion of the acid is passed into the trap to form a seal. Sufficient flow of air being maintained to prevent back travel through pressure, the digestion flask is heated gradually to about 300° C.; after being kept at this temperature for 2 hours, with frequent shaking, the contents of the flask are boiled for 5 minutes, the flame then removed and the air passed for 30 minutes to remove any silicon tetrafluoride from the system. The contents of the test-tube are boiled gently for 10 minutes with about 250 c.c. of water in a 500 c.c. Erlenmeyer flask, and then cooled to 50° C., the hydrofluosilicic acid being titrated with 0·1 *N* alkali in presence of phenolphthalein (1 c.c. corresponds with 0·0019 grm. of fluorine). After the titration, the solution is made acid with hydrochloric acid and tested for sulphate; if necessary, the sulphate is determined and the titration result corrected accordingly.

T. H. P.

Gum Tragacanth, W. Peyer. (*Chem. Zeit.*, 1924, **48**, 701.)—Gum tragacanth (*Astragalus gummifer*) is not a secretion, but is a pathological formation, probably due to external irritation by insects, etc., and oxidation by way of the injury. According to Tschirch, Hartwig, Rosenthaler and Runne, gum tragacanth does not contain oxidising enzymes, and although all forms of tragacanth give the guaiacum reaction, the author, in common with Oppenheimer, thinks this evidence uncertain; as also that of the pyrogallol reaction described in all the text books. The presence of oxidase in tragacanth mucilage (1:50) is indicated, however, by the addition of eserine, apomorphine, hydroquinone, phenolphthalein, aloin, guaiacum resin tincture, guaiaconic acid solution, guaiacol, α-naphthol, *p*-phenylenediamine, potassium iodide starch solution, with and without hydrogen peroxide or ethylhydroperoxide solution, α-naphthol- and dimethyl-*p*-phenylene-diamine. For testing gum tragacanth powder, the following substances are recommended in order of their utility:—Benzidine, adrenaline (1:1000), orcinol, gallic acid, resorcinol and leucomalachite green. In genuine tragacanth mucilage there are to be seen one or more spongy particles, often as large as lentils, which are dyed more or less intensely according to the reagent applied. These may be granules of the gum which become saturated with the solution of the reagent and dyed. Apparently some parts of the gum are richer in oxidase than others. Furfural is produced from the pentoses of tragacanth and reacts with orcinol, probably with the formation of orcein. It is uncertain whether resorufin or resacoin are produced in the resorcinol reaction.

P. H. P.

Japanese Saffron. E. Skarnitzl. (*Zeitsch. Unters. Nahr. Genussm.*, **1924, 48,** 213–217.)—Much of the Japanese saffron (*Crocus japonicus*) now on the market has been found to be largely adulterated with coloured barium sulphate and the leaves of the horse-thistle, *Onopordon acanthium*. The botanical and microscopical features of the latter are described in detail and illustrated. The more important diagnostic features are the large unicellular thick-walled bristles or hairs, which are shaped like a canine tooth and measure from 100–160μ by 30–38μ, and the short thickened trichome cells of similar dimensions attached to the large sclerenchymatous fibres. H. E. C.

Biochemical, Bacteriological, etc.

Chemical Constitution and Antiseptic Power. Th. Sabalitschka. (*Chem. Zeit.*, 1924, **48,** 703.)—The effect of various concentrations of different substances, with different hydrogen ion concentrations of the nutrient medium, on the development of *Micrococcus candicans*, *Bacillus coli*, *Sarcina flava* and *Penicillium glaucum* was tested, and the connection between chemical constitution and preservation effect was followed. Of the inorganic and aliphatic acids tried, formic acid had the strongest effect, a solution of 0·036 per cent. concentration being sufficient to prevent development of bacteria and fungi. The effect diminished in the following order:—Acetic acid, hydrochloric acid, sulphuric acid, maleic acid, malonic acid, fumaric acid, tricarballylic acid, suberic acid, phosphoric acid, malic acid, succinic acid, *d*-tartatic acid and glutaric acid. The inhibiting action of numerous cyclic compounds, such as benzoic acid, hydroxybenzoic acid, salts, esters and other derivatives of these acids, phenols, carbohydrates, etc., was also studied. Phenol, thymol and carvacrol caused the greatest effect, concentrations of 0·014 per cent. inhibiting the development of both bacteria and mould fungi. The methyl esters of benzoic acid, *p*-hydroxybenzoic acid, *m*-hydroxybenzoic acid, anisic acid, and cinnamic acid were effective at a concentration of 0·14 per cent., salicyclic acid and metachlorbenzoic acid at 0·21 per cent., and acetylsalicylic acid, and pyrocatechol at 0·28 per cent. Sodium benzoate only prevented development at a concentration of 1·5 per cent., and the sodium salts of the three hydroxybenzoic acids were not effective even at 2 per cent. The antiseptic action is increased by an alkyl hydroxyl group, decreased by a COOH-, HSO_3- and NH_2- group; halogen may either increase or reduce the action. The relative positions of the substituents have also a pronounced effect. Esters have, as a rule, a stronger action than free acids, salts a much weaker one. The pronounced action of the monophenols and benzoic acid depends not so much on the phenolic or carboxylic group as on the whole constitution of the substance. P. H. P.

Recent Research Work on Saponins. L. Kofler. (*Chem. Zeit.*, 1924, **48,** 702.)—An outline of previous work on the saponins is given. It is not clear whether saponins are absorbed through the intact intestinal walls, although this question is of fundamental importance for the explanation of therapeutic effects and for the toxicology of the saponins. An attempt to investigate this question by examination of the urine was made, but saponin drugs were found not to be serviceable diuretics, no saponin or sapogenin being detected in the urine in any single case. An indirect method of determining the approximate amount of saponins in the blood stream was based on the fact that they increase the amount of cholesterol in the serum. Intravenous injection of small quantities of primulic acid (a saponin from the primula) into rabbits, dogs and human beings produced in every instance an increase in the cholesterol in the blood serum, and this afforded a proof of the absorption of that saponin. Sarsaparilla root, used for centuries as a remedy against bubonic plague, was also found to increase the amount of cholesterol in the serum. P. H. P.

Quantitative Determination of Menthol Glycuronic Acid in Urine. A. J. Quick. (*J. Biol. Chem.*, 1924, **61**, 667–677.)—The quantitative methods for free and conjugated glycuronic acids are discussed and their defects pointed out. A new quantitative method proposed for menthol glycuronic acid depends on the extraction of the compound from urine with ether, subsequent hydrolysis of the conjugated acid with a dilute mineral acid, and finally a quantitative determination of the liberated glycuronic acid by means of either the Benedict or the Folin-Wu sugar method. Menthol glycuronic acid was chosen, since it can readily be isolated from urine and prepared pure so as to be suitable for a standard. It has the advantage of being easily soluble in ether and of being readily hydrolysed by dilute acids. It seemed almost impossible to find a general method for all conjugated glycuronic acids, but this method, specific for one, with modifications could probably be applied to others. The preparation of menthol glycuronic acid is described. A modification of the apparatus of the type designed by Clausen (*J. Biol. Chem.*, 1922, **52**, 263) was used for extracting the acid from the urine. A typical analysis is given which was made on a specimen of urine from a rabbit which had been fed with menthol. Modifications of the Benedict method which are necessary to make the method applicable to dilute reducing solutions are described. P. H. P.

Water Analysis.

Volumetric Determination of Total Carbonic Acid in Dilute Solutions of Calcium Hydrogen Carbonate or in hard Waters. E. M. Crowther and W. S. Martin. (*J. Chem. Soc.*, 1924, **125**, 1937–1939.)—Comparative figures are given for the following six methods of determination:—(1), (2) Addition of barium or calcium hydroxide alone; (3) and (4) of each with the addition of the corresponding chloride; (5) of calcium hydroxide and powdered calcium carbonate (to cause rapid "ageing" of the precipitate, as the freshly formed calcium carbonate is more soluble and more readily hydrolysed); and (6) of calcium hydroxide, calcium chloride and calcium carbonate. After standing for varying periods 0·5 *N* hydrochloric acid was used for the titrations (phenolphthalein as indicator), and the results show that the first three methods are unsatisfactory, the fourth only approximately accurate after 3 hours' standing, the fifth gives good results after 1 hour, whilst the most satisfactory is the sixth, which gives constant and accurate results after 10 minutes. D. G. H.

Agricultural Analysis.

Determination of the Ammoniacal Nitrogen in Soils. W. McLean and G. W. Robinson. (*J. Agric. Sci.*, 1924, **14**, 548–554.)—An extension of the Hissink method for the determination of ammoniacal nitrogen is given, which has the advantage of being independent of the water pressure in the laboratory. Twenty-five grm. of the soil, which is ground to pass a 3 mm. sieve, are wetted with 100 c.c. of *N* sodium chloride solution, allowed to stand for half-an-hour, and then filtered, and the soil is further washed with sodium chloride solution by decantation until 500 c.c. have been collected. The filtrate is distilled with magnesium oxide into 0·02 *N* sulphuric acid and titrated in the usual way, preferably with the use of the B.D.H. universal indicator carried to a distinct blue, but methyl red may be employed. Comparison is made with the results of aeration methods, and good results have been obtained, which suggests that the amount of ammoniacal nitrogen in a soil at any given time is definite and is not dependent on the conditions of the determination. H. E. C.

Determination of Nitrate and Ammonia in Nitrogenous Materials. O. M. Shedd. (*J. Agric. Res.*, 1924, **28**, 527–539.)—Strowd's method (*Soil. Sci.*, 1920, **10**, 333) has been modified by allowing the reaction to proceed for 24 hours in a flask fitted with a Bunsen valve before the final heating and distillation. The method gives good results and is applicable to the determination of small quantities of nitrate, such as occur in tobacco, or for the larger quantities present in fertilisers. The cold-water extract of the material is diluted to 300 c.c., fragments of pumice dust and of paraffin wax are added, and 2·5 to 3 grms. of sodium hydroxide, followed by 2 to 3 grms. of powdered Devarda alloy (Cu, 50; Al, 45; Zn, 5). After standing for 24 hours the liquid is heated slowly in the distillation apparatus for 2 hours, and then distilled into standard acid in the usual way.
H. E. C.

Organic Analysis.

Modified Dichromate Method for the Determination of Glycerol. H. B. Bennett. (*J. Chem. Soc.*, 1924, **125**, 1971–1975.)—In connection with work on the hydration of curd fibres of sodium palmitate the following modification of Hehner's dichromate method for the estimation of glycerol has been worked out, so as to render it applicable to the examination of 0·2 to 0·5 grm. in dilute solution with an accuracy of 0·1 per cent. To a clear solution obtained by warming the 10 grms. of the lye with 28 c.c. of water, 50 c.c. of 50 per cent. sulphuric acid are added, and after well cooling to complete the precipitation of the palmitic acid, a quantity of dried potassium dichromate about 0·1 grm. in excess of that required to oxidise the glycerol is added, and the liquid heated on a boiling water bath under a reflux condenser for 2 hours. The solution is then cooled and diluted to 1 litre, and 50 c.c. are withdrawn, diluted to about 600 c.c., and titrated with 0·1 *N* sodium thiosulphate. Glycerol is negatively sorbed by sodium palmitate at room temperatures, even in the absence of any salt. H. E. C.

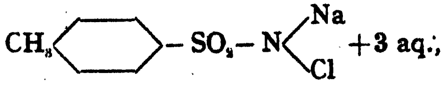
Studies on Pentose Metabolism. I. Colorimetric Method for the Determination of Furfural. (*J. Biol. Chem.*, 1924, **61**, 741–746.)—An accurate, simple and rapid colorimetric method is presented for the estimation of furfural, based on the colour produced with aniline and acetic acid. The red colour formed in the reaction readily detects 0·00001 per cent. and 0·00004 per cent. of furfural can be determined quantitatively with an error never greater than 6 per cent. The red colour is not produced if the aldehyde group of the furfural molecule is destroyed, as, for example, by oxidation to pyromucic acid or reduction to furfurol. A table shows the comparison of the colorimetric method for furfural with the gravimetric phloroglucinol (*U.S. Dept. Agric., Bureau of Chem., Bull.* 107 (rev.), 54) method, and Pervier and Gortner's electrometric method (*J. Ind. and Eng. Chem.*, 1923, **15**, 1167, 1255). Full experimental details are given. The reaction of furfural with aniline acetate is specific for furfural, and thus may not only be valuable in studying the decomposition of pentose-containing substances, but, owing to its sensitiveness, is also applicable to the study of the furfural-producing substances occurring in the blood, body fluids and urine. P. H. P.

Inorganic Analysis.

Colour Reactions of Cryogenine with the Heavy Metals. L. Bornet. (*J. Pharm. Chim.*, 1924, **30**, 356–358.)—A freshly prepared saturated aqueous solution of cryogenine with a few extra crystals of the solid, is shaken with the solution of the metallic salt; one or two drops of hydrogen peroxide may be added to increase the sensitiveness of the reaction, which should take place in neutral solution. Copper salts give intense red colorations proportional to the amount present. Mercury salts give pink colorations followed by violet precipitates, but the solution must be exactly neutral, and the addition of a few c.c. of hydrogen peroxide should be followed by an acetate; if benzene or ether are then added the precipitate collects as a violet band at the zone of contact of the liquids. With iron salts a red-brown coloration is seen, and in the case of ferric chloride the reaction is as sensitive as with salicylic acid. Strongly (hydrochloric) acid solutions of chromates or of bichromates added to the powdered cryogenine give wine-red colorations.

D. G. H.

Chloramine as a Substitute for Iodine in Analytical Work. A. Noll. (*Chem. Zeit.*, 1924, **139**, 845.)—The compound *p*-toluene-sulpho-choramide-sodium



$$\mathrm{CH_3}\langle\ \rangle\mathrm{-SO_2-N}\genfrac{}{}{0pt}{}{\mathrm{Na}}{\mathrm{Cl}} + 3\ \mathrm{aq.},$$

generally known as chloramine, is stable when crystallised from boiling water;

it has the property, when dissolved in cold water, of decomposing potassium iodide solution. In 0·1 *N* solution it keeps well in a dark glass bottle, and can be standardised before use on 0·1 *N* arsenious acid or sodium thiosulphate solutions. Chloramine is much cheaper than iodine, a 0·1 *N* solution costing only about a fortieth of the price of a 0·1 *N* solution of iodine. Tables are given to show the close agreement between results obtained by using chloramine, as compared with iodine, in analysing calcium bisulphite liquors, sodium bisulphite solutions of varying concentrations, and in determining sulphur dioxide in pyrites gases.

R. F. I.

Electrolytic Separation of Lead and Tin. A. Lassieur. (*Compt. rend.*, 1924, **179**, 827–829.)—The solution, containing less than 0·15 grm. of tin per 100 c.c., is treated with one-tenth of its bulk of strong hydrochloric acid, one-twentieth of strong hydrofluoric acid, and one grm. of hydroxylamine hydrochloride. It is electrolysed at 60° C. for 10 minutes, then for another 10—15 minutes while the electrolyte is being cooled to room temperature (0·4 volt.). Lead is deposited, whilst tin forms a fluoride complex provided it is present in the stannic condition. If it is not present in that condition, the solution is boiled for a few minutes with 5 c.c. of hydrogen peroxide (12 volumes). After removal of the lead, the solution is treated with 10 grms. of boric acid and 10 grms. of ammonium oxalate, diluted to 300 c.c., warmed, and electrolysed for tin at 0·6 to 0·65 volt with a copper-coated cathode.

W. R. S.

Separation of Rare Earths from Uranium. Alkali Uranylsalicylates. G. Canneri and L. Fernandes. (*Gazz. Chim. Ital.*, 1924, **54**, 770–773.)—The fact that salicylic acid forms the complex uranylsalicylic acid with uranium, whereas it does not unite with salts of the rare earths to give soluble complexes, furnishes a means of separating these earths from uranium. The hot, faintly acid solution is treated with excess of salicylic acid, and ammonia solution is then added, drop by drop, until the liquid assumes a blue colour, but still remains acid. A slight excess of concentrated ammonium oxalate solution is next added, the liquid being afterwards boiled, left for a few minutes to deposit the precipitate, then filtered, and the precipitate thoroughly washed with boiling ammonium oxalate solution; the rare earth oxalate thus separated is calcined and weighed. The solution containing the uranium is acidified with concentrated hydrochloric acid, allowed to cool, and filtered to eliminate most of the salicylic acid, which is washed with cold water. The filtrate is evaporated to dryness with nitric acid, and the residue calcined on a sand-bath to expel organic matter, and dissolved in hydrochloric acid. Treatment of the solution with ammonia solution, drop by drop, results in the precipitation of ammonium pyrouranate, which is dealt with in the usual manner.

T. H. P.

Sensitive Colour Reaction for Magnesium. F. L. Hahn, H. Wolf and S. Jäger. (*Ber.*, 1924, **57**, 1394–1396.)—A sensitive rest for magnesium is based upon the fact that it produces a bright blue coloration or blue precipitate in an alcoholic solution of 1.2.5.8-tetrahydroxy-anthraquinone. The reaction is capable of detecting 0·001 mgrm. of magnesium, but is not so sensitive when ammonium salts or phosphates are present. In applying it to the detection of minute quantities of magnesium in aluminium an excess of sodium hydroxide or of tartrate and alkali is used to keep the aluminium in solution.

Separation of Molybdenum from Tungsten. I. Koppel. (*Chem. Zeit.*, 1924, **48**, 801–802.)—The solution of alkaline molybdate and tungstate is almost neutralised with formic acid and treated with a few c.c. of ammonium sulphide followed by 5 c.c. of formic acid, the total bulk being about 100 c.c. The solution is heated for some time on the water-bath to cause coagulation of the precipitated molybdenum sulphide. This is collected on a Gooch crucible, washed with water containing a little formic acid, and weighed as sulphide, or carefully roasted to trioxide. The filtrate is evaporated with sulphuric acid for the precipitation of tungstic acid. W. R. S.

Precipitation of Tantalum and Niobium by "Cupferron," and their Separation from Iron. H. Pied. (*Comptes rend.*, 1924, **179**, 897–899.)—Tantalum and niobium are precipitated quantitatively from their oxalo-tartaric solution containing sulphuric acid by "cupferron," the method of separating iron indicated by Weiss and Landecker (*Zeitsch. anorg. Chem.*, 1909, **64**, 69) being thus applicable. The freshly-precipitated hydroxides are dissolved in oxalic acid solution, and the liquid then treated with a little tartaric acid, warmed gently, treated with ammonium hydrosulphide, and left overnight. After removal by filtration of the ferric sulphide, which is quantitatively precipitated, the liquid is treated with excess of sulphuric acid and freed from hydrogen sulphide by boiling, and from sulphur by filtration. The cold solution is mixed with "cupferron" and shaken vigorously, the appearance of a whitish film on the surface indicating the end of the reaction. The precipitate is immediately filtered off with the aid of slight suction, washed with very dilute sulphuric acid, partly dried, and carefully calcined. The tantalum and niobium, and any titanium present, are precipitated completely in this way.

The oxalate solution may be obtained by direct treatment of the fusion mass given when the calcined oxides and many minerals are attacked by pyrosulphate. The rare earths and thorium do not hinder the solution, but form a crystalline precipitate, the extent of which indicates the proportion of these constituents. Further, titanium may be determined in the solution, since oxalic acid has no action in the cold on the coloration given with hydrogen peroxide. T. H. P.

Examination of Sodium Dithionate. F. M. Litterscheid and H. Löwenheim. (*Chem. Zeit.*, 1924, **48**, 881–883.)—One grm. of the salt, on being heated in a closed tube, should only give off water and sulphur dioxide without production of a sublimate (sulphur); the residue should remain white. The quantitative analysis may be carried out very simply by heating the finely-powdered salt in a porcelain crucible at 60° C. for 1½ hours, then at 100° C. for

½ hour. The loss in weight represents water of crystallisation; the salt has the formula $Na_2S_2O_6.2H_2O$, but a hexahydrate is also known. The dry substance is then gently ignited: $Na_2S_2O_6 = Na_2SO_4 + SO_2$. Decomposition takes place at 250° C. After half-an-hour's heating the residue is left to cool in a desiccator and weighed as Na_2SO_4, giving SO_2 by difference. The residue may serve for the estimation of SO_3, or for tests for carbonate, chloride, etc. A process is also described in the paper, in which the dehydrated salt is heated in a glass tube and the evolved sulphur dioxide absorbed in standard iodine solution; it offers no advantage over the simpler method described above. W. R. S.

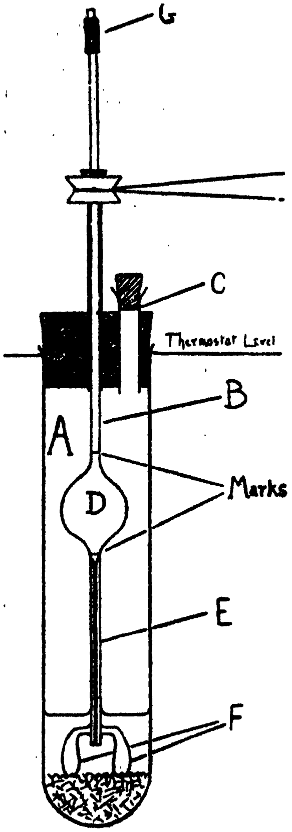
Physical Methods, Apparatus, etc.

Measurement of P_H Value of Dairy Products by Billmann's Quinhydrone Electrode. V. Lester. (*J. Agric. Sci.*, 1924, **14**, 635–641.)—Billmann's quinhydrone electrode (*cf. J. Chem. Soc.*, 1923, **123**, 2203) affords a simple and rapid method for the determination of P_H in milk, cream and whey, and is of special value because methods depending on dialysis are unreliable and the platinum-hydrogen electrode method is slow and open to many sources of error. The method is applied by adding 50 mgrms. of quinhydrone to 10–20 c.c. of the sample and placing in position three smooth gold electrodes, the connecting link with the standard quinhydrone electrode being the usual potassium chloride bridge. The standard consists of three platinum or gold electrodes in a saturated quinhydrone solution containing 0·01 *N* hydrochloric acid and 0·09 mol. potassium chloride, and a siphon tube from this dips into an ordinary potassium chloride electrode vessel. The potential becomes constant within 5 minutes, and $P_H = 2{\cdot}03 + \frac{\pi}{0{\cdot}0577}$, where π is the potential difference which has been measured. H. E. C.

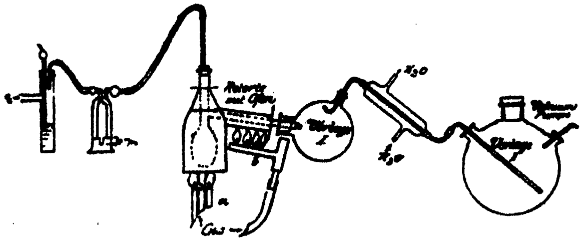
Microscopical Examination of Cacao Products. V. A. Pease. (*J. Ass. Off. Agric. Chem.*, 1924, **8**, 176–178.) In the microscopical examination of cacao products, and especially if a slide is to be kept for some time, the standard 1:1 chloral hydrate clearing solution used as mounting medium may be replaced with advantage by Hertwig's solution, composed of 25 c.c. of dilute hydrochloric acid (1:8), 10 c.c. of glycerin, and 45 grms. of chloral hydrate. Care must be taken to use just enough of the solution to fill the space under the cover glass, as the glycerin becomes more fluid when warmed and, if excess is employed, may carry fragments of tissue beyond the edge of the cover-glass. The advantages are that drying of the mount and formation of crystals under the edge of the cover-glass are avoided, and that the procedure is quickened, as the preparation may be made, warmed for about 20 minutes, and counted after standing for only 30 or 45 minutes.

T. H. P.

Apparatus for the Viscometric Determination of Transition Points. N. H. Hartshorne. (*J. Chem. Soc.*, 1924, **125**, 2096–2099.)—The apparatus illustrated enables successive determinations to be made in the same vessel at different temperatures. The tube A is 25×6 cm. and carries the combined viscosimeter and stirrer B and a closed tube C. The bulb D has capacity about 20 c.c. and the capillary E is 10 cm. in length, and of diameter about 0·75 mm. F is a glass paddle sealed on to the capillary. For the determination 80 c.c. of the solution together with excess of the salt in A are immersed in a thermostat and rotated until equilibrium is reached, then the salt is allowed to settle, the dust cap G is removed and the solution is sucked up above the higher mark and the time of flow from the upper to the lower mark is noted as usual. By means of additions or withdrawals through C the quantities of solid and solution can be adjusted. H. E. C.



Sublimation Apparatus. A. Gutbier and T. Payer. (*Chem. Zeit.*, 1924, **133**, 807.)—The apparatus described has the advantage of being suitable for the sublimation of compounds of high volatility, or of those containing water or acid from which it is difficult to separate them, or of those sensitive to decomposition. It consists mainly of a hard glass retort in the form of a distilling flask enclosed in a small fireclay hood, the bottom of the flask being protected by asbestos paper. The side-tube of the retort is closely wrapped in thin platinum foil, passes through a cylinder of fireclay and delivers into two receivers of glass, I and II, which are connected by a small condenser. Receiver II has tubular openings, one to



facilitate cleaning and one connected with a water-pump which draws air or other gas through the whole apparatus. Where necessary, the joints are made with a cement of asbestos and sodium silicate. The fireclay cylinder around the side-tube only extends 50 mm. (as shown in diagram), its end being provided with an asbestos disc to protect receiver I from undue heat. The gas supplied to the apparatus during a sublimation passes through a glass vessel which acts as a safety-valve at the desired pressure, thus avoiding liability to "blow-out" as a result of possible blocking of the tubes. The delivery tube into the retort is bent at its end to conform to the side tube, thus tending to remove water vapour and any decomposition-products and to carry them over into receiver II. The internal pressure should be maintained between 30 and 40 mm. of water, the surface of the water in the safety valve being covered with vaseline-oil to prevent water-vapour being carried over. The sublimation of selenium oxide is taken as an example and described in detail. By this method the retort is left quite clean at the end of the process. R. F. I.

Reviews.

CHEMISTRY IN THE TWENTIETH CENTURY. AN ACCOUNT OF THE ACHIEVEMENT AND THE PRESENT STATE OF KNOWLEDGE IN CHEMICAL SCIENCE. Prepared under the guidance of a Committee with Dr. E. F. ARMSTRONG, F.R.S., as Chairman and Editor. Pp. viii.+281. London: Ernest Benn, Ltd. 1924. Price 15s. net.

As to the value which may accrue from the Chemical Exhibit at the British Empire Exhibition at Wembley there may be differences of opinion. As to the desirability of publishing such a work as the one now in our hands there can be no possible difference of opinion. It may not have been one individual who originated the idea; but whether individual or committee we are thankful that the idea has materialised in such an acceptable form. The essayists are all distinguished men of science who have very judiciously put into the forefront the achievements of British chemists without laying any invidious stress upon nationality, and who have not restricted too narrowly the time limits of their theses. The volume, we are told, has been compiled for those "who have received, or are receiving, a training in science." It was, therefore, important that the writers should have a due regard to the historical setting of their subjects. Most of them have succeeded in furnishing a properly balanced background for their pictures, and will thus capture and hold the attention of the older chemists as well as interest and instruct the newer generation.

As this volume must make an appeal to all the readers of THE ANALYST, we think we ought to make them acquainted with the diversity of its contents and the authors of the monographs. These are as follows:—"The Rôle of Chemistry in Physical Science" (Prof. J. I. O. Masson), "The Structure of the Atom" (Dr. E. N. da C. Andrade), "Crystallography" (Sir Hy. A. Miers and Mr. T. V. Barker), "X-ray Analysis of Crystals" (Sir W. H. Bragg), "The Rare Gases of the Atmosphere" (Dr. M. W. Travers), "Chemistry of the Carbon

Compounds" (Prof. J. F. Thorpe), "Milestones in Organic Chemistry" (Prof. H. E. Armstrong), "Chemistry of Colloids" (Dr. Wm. Clayton), "Catalysis" (Dr. T. P. Hilditch), "Fats and Oils" (Mr. John Allan), "Sugars and Carbohydrates" (Dr. J. C. Irvine), "Cellulose" (Mr. C. F. Cross), "Colour in Nature" (Mr. R. Furness), "Coal Tar Colours" (Mr. E. A. Bearder), "Syntheses in the Terpene Series" (Professor G. G. Henderson and Mr. Alex. Robertson), "The Alkaloids" (Prof. F. L. Pyman and Dr. T. A. Henry), "Nitrogenous Constituents of the Living Cell" (Dr. R. H. A. Plimmer), "Biochemistry and Fermentation" (Prof. Arthur Harden), "Chemistry in Agriculture" (Sir John Russell and Mr. H. J. Page), "Alloys" (Prof. C. H. Desch), "Pottery and Refractories" (Mr. Joseph Burton), "Flame, Fuel and Explosion" (Dr. H. F. Coward), "Explosives" (Sir R. Robertson), "Chemistry of Photography" (Sir W. J. Pope and Mr. Walter Clark).

The Introduction has been written by Dr. E. Frankland Armstrong, who directs attention to the clear picture the first few articles give of the development of modern theories of chemistry, as well as to the specialised articles which illustrate, with what he calls "kaleidoscopic rapidity," the progress which has been made in certain industries by the application of the new theories.

The articles by Professor Masson and Dr. Andrade will be read with great interest, because they reveal the great change that has taken place in the relations of the chemist and the physicist. As Dr. Armstrong points out, the atom is no longer hypothetical—it is a real object of definite size and shape, and Professor Masson and Dr. Andrade demonstrate this fact in such a succinct and lucid manner that no chemist can fail to understand its significance. The diagrams representing the orbits of the electrons are very useful.

The history of chemistry acquires from the hand of Dr. Morris Travers a chapter which is of the greatest value. His work, in conjunction with Sir Wm. Ramsay, upon the rare gases of the atmosphere, has drawn together the experiments of Henry Cavendish in the eighteenth century and the discovery of radium with its emanation (niton) at the end of the nineteenth.

Another monograph which will be read in conjunction with those just mentioned is the one by Dr. William Clayton on "The Chemistry of the Colloids." This subject is peculiarly interesting, because the initiation of this branch of chemistry was in Thomas Graham's papers on "Liquid Diffusion," and may be legitimately regarded as of British origin. Dr. Clayton has much to say about the practical side of investigations in colloidal chemistry in respect to their application to soaps, dyes, paints, "smokes," dust explosions, etc., etc.

The two articles on crystallography are specially worthy of notice, for not only are they written by the most eminent authorities in that branch of science, but also, as Sir Henry Miers says, the contributions to it by British investigators have been of the highest importance; moreover, they are full of information and well illustrated.

Space does not permit of particular mention of those essays of a more specialised character. They are necessarily more restricted in their appeal than those named above, which cannot fail to kindle anew in every chemist the enthusiasm for his science which may have faded somewhat in the course of laboratory practice. Perhaps Dr. Armstrong really meant all that the word "kaleidoscopic" should signify. In any case, the beauty of the ordered progress of the discovery of the constitution of matter shines forth in such a way as to impress the most callous reader. That this country has made so great a contribution to it is a cause for thankfulness. The volume is well printed, well bound and really most moderate in price. WILLIAM KIRKBY.

MIKROCHEMISCHES PRAKTIKUM. By FRIEDRICH EMICH. Munich: J. F. Bergmann. 1924.

The award of a Nobel prize in 1923 to Prof. Pregl in recognition of his improvements in micro-chemical technique may help to remove much of the prejudice against working with small quantities of material where circumstances do not compel their use. This prejudice is very natural considering the instinctive preference of chemists for methods in which the mechanical difficulties are so much less, and the limits of error more accurately known. Howevet that may be, it is certain that the ingenuity and simplicity of the devices described in the pages of this little book are such that it cannot fail to appeal to a much wider circle, and receive far greater consideration than has been accorded to the great majority of earlier works. It may not be uninteresting to recall in passing that one of the first comprehensive treatises dealing with micro-chemical methods was that of Theodore G. Wormley, which was published as far back as 1867.

Prof. Emich, who is well known as one of the leaders in this branch of work, has produced a book which will certainly prove of considerable value in suggesting ways of avoiding the disappointment of an incomplete investigation by reason of scarcity of material.

An admirable general section describes in considerable detail the manipulative side of the work, giving numerous illustrations of the simple apparatus used, and dealing with the general considerations necessary in the reduction of ordinary laboratory methods to a micro-chemical scale. The application of the methods is dealt with in the second part, and a great many examples are given, chiefly intended as exercises in inorganic and organic analysis and organic preparations.

The author lays the greatest stress on the need for patient practice, and urges the repetition of every typical reaction until positive results are as certain as on the ordinary scale. The need for experience and confidence will be very evident when it is seen that in qualitative tests the amount of material in question may be of the order of one-thousandth milligramme, and that the quantitative methods described are usually sensitive enough to be reliable when working with 2 to 10 milligrammes of material.

The adaptation of the ordinary physical determinations, such as melting and boiling points, refractive indices, molecular weights, etc., where micro-chemical methods are so often called for, is adequately described.

Prof. Emich has written a most helpful and suggestive little book, and one which may be warmly commended to the attention of chemists, the great majority of whom—at one time or another—find themselves compelled to work with very small quantities of material, and who might, perhaps, with advantage, make more frequent incursions into this field of chemical procedure. The book is clearly written, the illustrations are numerous and good, and, unlike many of the works which are being published in Germany at the present time, it is printed on reasonably good paper. A. CHASTON CHAPMAN.

A SYSTEMATIC HANDBOOK OF VOLUMETRIC ANALYSIS. FRANCIS SUTTON. Eleventh edition. Edited by W. L. SUTTON, F.I.C., and A. E. JOHNSON, B.Sc., F.I.C., A.R.C.Sc.I. Pp. xii.+629. London: J. and A. Churchill. 1924. Price 35s. net.

After a long interval of thirteen years, a new edition of this, the oldest treatise on volumetric analysis in the English language, has made its welcome appearance. Few, if any, British chemists engaged in analytical practice have not, at some time or other, had an opportunity to thank "Sutton" for useful advice on a problem outside their daily routine, or for obtaining reliable information on a volumetric process new to them. Considerable changes have been made in the present work; the section on Gas Analysis has been re-written by Mr. H. Hollings and Dr. J. S. G. Thomas: it occupies 103 pages, and is indexed separately. The editors describe it as "a comprehensive and up-to-date little treatise on the subject such as has not previously existed in English." Part of the section is devoted to information not commonly found in analytical handbooks, *e.g.*, the preparation, graduation, and calibration of gasometric apparatus. The chapter on the calibration of graduated instruments comprises a tabulation of the limits of error allowed by the National Physical Laboratory.

The Section on Indicators is "entirely re-written, with many additions"; it is, therefore, disappointing to find that the question of hydrogen ion concentration, which has been the subject of so much investigation in recent years, has been ignored. No indication is given of the P_H range of indicators; no "universal" or mixed indicators are described. Again, methods of, and apparatus for, potentiometric and conductometric titration find no place in the book. The reviewer believes he is not guilty of harsh criticism when he says that the omission of the above subjects constitutes a failure to keep the book up to date.

In the sections dealing with Applied Methods of Analysis, the text-matter might be enriched here and there with the description of, or, at least, a reference to, some more recently published processes. Thus, in present-day works practice, the favourite method for cerium is Metzger's bismuthate process. An interesting contribution to the volumetric estimation of aluminium has been made by Kraus (ANALYST, 1922, **47**, 92). Cadmium—as well as some other divalent metals—can be estimated alkalimetrically as phosphate (ANALYST, 1916, **41**, 124). The chapter on arsenic might with advantage include a description of its estimation in ores by a process of conversion into arsenate and titration of the latter with iodide and thiosulphate (Low, 7th edition, 1914, 45–48). Sodium hydrosulphite (hyposulphite), $Na_2S_2O_4$, is used in large quantities as a bleaching and reducing agent ("hydros"), and its analysis (ANALYST, 1922, **47**, 369) should be included in a work of this character. The best method for the removal of tungstic acid in the volumetric assay of tin ores is that of Powell (*J. Soc. Chem. Ind.*, 1918, 285). For the reduction of titanium solutions the use of Jones' reductor is a decided advantage (ANALYST, 1924, **49**, 153). Under vanadium, a method for its estimation in ores—including reduction by sulphur dioxide and oxidation by permanganate—might have been added. In most, if not all, Continental zinc smelting works, glazed lead carbonate paper is used as an external indicator in the Schaffner method.

A multiplicity of procedures for the titration of one and the same element is sometimes evident. The use of some of these methods should be discouraged, as they are of doubtful value, and their place has been taken by more accurate ones. For example, the chapter on Copper comprises ten methods, some with modifications. In his moderately wide experience of the metallurgy of copper, the reviewer has seen only the four following methods used, in addition to electrolysis: the iodide, the cyanide, a modification of the thiocyanate, and the colorimetric method. If the other methods are rarely used, they must have been found less convenient or accurate, hence the wisdom of keeping them in the book may be questioned; this applies particularly to Pelouze's process. The text-matter on mercury, arsenic, and antimony presents an almost embarrassing choice of alternatives.

The work appears very free from misprints: the term "Dihydroxytartaric" (for "Dihydroxytartrate") appears in the title on p. 65, and printer's slips were observed in pages 356, 419, and 512. A manifest error in nomenclature has managed to evade revision, one of the solutions used for certain acidimetric titrations being called "Ammonium=Copper Solution" (p. 56). Whatever nomenclature is applied, the compound $CuSO_4.4NH_3.H_2O$ contained in that solution is certainly not an ammonium salt. Those who still hesitate to employ Werner's nomenclature ("tetramminecupric sulphate") have the alternative of referring to this reagent as "ammoniacal copper solution." The time-honoured appellation "Alkaline Earths" has been changed in this edition to "Alkali Earths" (p. 73); in the reviewer's opinion this is hardly an improvement, as "alkali earth" should, like "alkali waste," refer to some kind of material containing alkali in admixture.

The Atomic Weight Table used in this book is that of 1923, in which the value for antimony is still given as 120·2. The figure 121·8 (ANALYST, 1924, **49**, 49, 152) will probably be adopted in the next International Table. Meanwhile it is advisable to standardise solutions used in antimony titrations against the pure metal or, if Sb_2O_3 is required, against the pure oxide.

W. R. SCHOELLER.

QUALITATIVE ORGANIC ANALYSIS. AN ELEMENTARY COURSE IN THE IDENTIFICATION OF ORGANIC COMPOUNDS. By OLIVER KAMM. Pp. 260+vii. New York: John Wiley & Sons, Inc.; London: Chapman & Hall. Price 12s. 6d. net.

In this book, which is based on S. P. Mulliken's "Identification of Pure Organic Compounds," there is developed a system of qualitative organic analysis which is somewhat analogous to the group system which is almost universally used in Inorganic Chemistry. The solubility of compounds in several common solvents, together with the fact of homology form the basis of the scheme.

Part A of the book gives a brief outline of the steps involved in the identification of organic compounds, and later the question of solubility is discussed, and certain approximate rules for the prediction of this property are deduced. This is followed by a very useful section on classification reactions, most of the common types being treated fully.

Part B, entitled "Laboratory Directions," commences with an account of the usual procedure for the examination of a single substance, and a number of exercises on class reactions are worked out. The importance of the tests for characteristic elements and the deductions therefrom are singularly overshadowed by the less helpful solubility discussion, and many an unwary student may be trapped in consequence. In passing, it may be pointed out that the standard nitroprusside test for sulphur is not mentioned in the text. The chapter on derivatives is of special merit, but it is curious that no reference is made to the important art of crystallisation. Lack of skill in this operation surely prevents physical constants from being determined, and hence final identification becomes impossible. The section closes with a chapter on the treatment of mixtures.

Part C concludes the book and contains the physical constants of 2000 or more compounds classified in ascending order of melting point (or boiling point) according to the solubility groups which are the basis of the scheme of analysis.

Many books, most of very limited usefulness, have been written with the object of helping students to identify organic compounds. All that the best books attempt to give is only a series of general hints to enable class assignment to take place, leaving the greater part of the process to the student's knowledge of theoretical chemistry—and to his wits. This is the utmost any book on the subject should do, for the object of organic "spotting" is to get students to increase their knowledge of organic chemistry by continual reference to the literature of the subject. The present book is perhaps too comprehensive, and a slavish adherence to its scheme will take from qualitative organic analysis much of its value, and at the same time render the process rather tedious. Nevertheless, those working for B.Sc. Hons. and A.I.C. (in organic chemistry) will find the book useful, provided that it is regarded merely as a help and not as an infallible guide.

HAROLD TOMS.

THE CHEMISTRY AND PHYSICS OF CLAYS AND OTHER CERAMIC MATERIALS. By ALFRED B. SEARLE. Pp. xiii.+695. London: Ernest Benn, Limited. 1924. Price 55s.

This book comprises fifteen chapters, the first seven of which deal with the structure, strength and other physical and mechanical properties of ceramic materials and with the changes effected in them by the addition and removal of water. Two chapters are devoted to the chemistry, one to the mineralogy, and one to the physico-chemical reactions of the materials. The remaining chapters embrace thermal, electrical and optical properties.

As pointed out in the preface, although craftsmanship in the ceramic industries had reached a high degree of perfection in very early times, the application of scientific methods to them is comparatively recent, and has been most marked during the last decade. Although this general statement is true, it must not be forgotten that notable attempts to apply scientific methods to the problems of ceramics have been made in the past, as the work of such leaders as Wedgwood, Brongniart and Seger abundantly shows. Further, it is also stated that the volume is not intended to be exhaustive, but to provide a description of the properties of various ceramic materials and the application to them of the more important principles of chemistry and physics, "as will be equally useful to students, manufacturers and users," and the question naturally arises, whether it is possible or even advisable, to attempt to meet the requirements of three such different classes of readers. Be that as it may, much of the information in the book ought to be quite unnecessary, say, for the student of chemistry, and at the same time would be insufficient for the manufacturer to acquire that knowledge of the fundamental principles of the science that he ought to possess in order to follow the subject intelligently.

The book has certain drawbacks. In places the views held by different authorities on some point—views often diametrically opposed—are given as bare statements without any discussion of the arguments in favour of either. As an instance may be mentioned the statement of the views of Rohland, Schurecht, and Bleininger and Fulton on the effect of alkalis and acids on the quantity of water required to develop plasticity (pp. 269–270). Another drawback which seriously hampers the use of the book for reference is that a subject is briefly mentioned, and then for fuller information on it the reader is referred to later pages or chapters. As an illustration of this, colloidal substances are mentioned on p. 7 and p. 9, and on both the same words occur, *viz.* that these substances "are described more fully on page 10 and in Chapter VI." To borrow a simile from the nomenclature of colloids, the book would appear to be an effective dispersion medium. This lack of arrangement leads to much repetition and between pages 400 and 405, two other books by the same author are referred to ten times, apart from other places.

Readers of THE ANALYST will probably be attracted most by the chapters dealing with the chemical constitution and physico-chemical reactions of ceramic materials. In them chemists will find a good deal with which they are familiar, but unless they are already conversant with the specialised branch of chemistry that deals with silica and its compounds, the book will not be easy reading or prove to be a convenient introduction to the subject. At the same time the chapter contains several interesting phase-diagrams, and the section on corrosion should be useful to those who are unfamiliar with the conditions that influence resistance to corrosive action.

Unfortunately the chapter on the optical properties of ceramic materials is short and hardly lays sufficient emphasis on this valuable but very difficult branch of the work.

Within the compass of 700 pages the author has courageously attempted the survey of a wide field and has brought together a large amount of useful information. There are more references to original papers than in some other books by the same author, but the inclusion of still more would have been a great assistance to the student of the subject. Obviously omissions must occur, but in discussing the evolution of the formulæ that have been proposed to connect chemical composition with refractoriness, the omission of any mention of Richter's Laws seems to spoil the continuity of the argument, and in the chapter on drying there is no reference to the work of Aron or of Jackson and Rich. Unfortunately, in places the wording is rather ambiguous; for instance, on p. 43, corundum and carborundum might be easily confused, and on p. 17 no clear distinction is drawn between the use of sillimanite as a raw material and sillimanite crystals produced by the action of heat in articles made from argillaceous materials.

The book has a copious index, is clearly printed and is in an attractive form, except that, to the writer, the pages seem too wide for easy reading. There are very few misprints; on p. 436, "ammonium cyanite" should of course be "ammonium cyanate," and unfortunately, the author of the well-known book on refractories is spoken of as "Harvard" instead of "Havard."

In conclusion, the perusal of the book leaves the impression that the theme has not been developed as logically as it might have been, and, though an amount of useful information has been presented in a convenient form, it is advisable to preserve a critical attitude with regard to many of the points.

WALTER C. HANCOCK.

TECHNICAL WRITING. By T. A. RICKARD. 2nd Edition. Pp. ix.+337. New York: John Wiley & Sons, Inc.; London: Chapman & Hall. Price 10s. net.

It is not enough to have something to say; even a technical report may be marred by the use of the wrong words or by their bad arrangement. This is essentially the text upon which is based a critical discussion on the writing of technical English. The general scope of the book, which has deservedly reached its second edition, may be gathered from the headings of some of its seventeen chapters, which include: Naturalness, Clearness, Precision, Superlatives and the Superfluous, Slovenliness, Jargon, The Wrong Word, Construction, Composition, and Style.

The chapter on Punctuation deserves especial mention, for although the author dissents in some respects from the more commonly accepted conventions, he makes out a convincing case for his suggested usage.

In his section on the use of the subjunctive he does not take up the extreme position of the authors of "The King's English," who say: " 'Were' is often right and almost always necessary; other subjunctives are never necessary, often dangerous, and in most writers unpleasantly formal." The author of this book, however, remarks "the subjunctive is a mark of scholarship; it is ignored by those who are not particular in such matters, and unfortunately it is a source of perplexity to those unfamiliar with the use of it."

Probably the best rule of guidance over this and similar debatable ground is to use the older forms until general custom makes it pedantic to retain them. Few writers of modern English would now use the expression "it *were* useless to protest"; it has an archaic sound. Some forms of the present subjunctive are nearing the same stage, if they have not already reached it.

The same practical rule of wide acceptance may be applied to the modern extension of the use of an active participle with a passive verb. Although the phrase "speaking generally" and some others have secured the sanction of custom, is there as yet any sufficient justification for the sentence: "The liquid *is titrated, using* phenolphthalein," which is so frequently seen in both English and American chemical journals? What is the objection to "*being used*"?

Some changes in the use of words are inevitable, and in time one must conform to them. It may, perhaps, even be necessary to acquiesce in the local mutilation of words, though one cannot help slyly wondering what reply the author would have made to Archdeacon Hare, who in the fifties of the last century expressed a hope that "such abominations as *honor* and *favor* would henceforth be confined to the cards of the great vulgar."

The author strongly condemns the use of long and abstruse words where the same meaning is conveyed by simpler ones. We are only slowly abandoning this legacy from those who upheld the tradition of Dr. Johnson, and turned *men* into *individuals*, *places* into *localities*, and *homes* into *residences*, and it is not easy to avoid taking our share of it; even the author of this book has not escaped, for more than once he speaks of "aberrances." Possibly that ugly word has a shade of meaning not conveyed by "lapses," but it would have been worth some sacrifice of precision to avoid its use.

In the chapter on Jargon the writer of scientific papers is warned against applying trite phrases such as "the swing of the pendulum" or "a bolt from the blue." This, again, is not always easy, for memory unconsciously reproduces set groups of words. In his remark that "these outworn figures of speech" are "taboo to the intelligent writer," the author himself approaches very close to his forbidden ground. A well-known writer, long since dead, once gave me the same advice in still more terse and forcible words: "Shun the cliché," he said, "as you would the devil."

Throughout the book numerous quotations are given to show how things should not be written and the reason why. Most of these are drawn from metallurgical and geological sources; had the author referred also to chemical publications he would have found a richer store. Our old friends, "data *is*" and "phenomena *is*" are ridiculed. They are of American origin, but we can match them with "policeman" used as a verb ("policemanning it"); and such gems as "bring to the boil" and "let stand" are shared impartially by chemists in either country.

Our language is the heritage of all the English-speaking peoples. Sometimes Americans take exception to criticisms of innovations that they have brought into it. But, as the author, himself an American, remarks (p. 90): "The official language of the United States, a language that originated in Great Britain, is written in the same way by those who write it well on either side of the Atlantic. I can assure you that Mr. Eliot and Mr. Butler, for example, speak and write the same language as Mr. Asquith and Mr. Balfour*; and if any of us speak or write differently from them, we may be using British or we may be using American, but we are not using English."

One cannot be too grateful for the effective plea on behalf of good English made in this small book. The author is impartial in his comments, which are full of humour and sound sense, and the rules he gives will well repay close study. It is a book to keep ready for constant reference.

EDITOR.

* Uncorrected from 1st Edition, published in 1916. --EDITOR.

Publications Received.

A SYSTEM OF PHYSICAL CHEMISTRY. Vol. III. Quantum Theory. By W. C. McC. Lewis, D.Sc. Pp. 407. London: Longmans. Price 15s. net.

SYNTHETIC ORGANIC COMPOUNDS. By S. P. Schotz, D.Sc. Pp. 412. London: Ernest Benn Ltd. Price 45s.

PHYSICAL CHEMISTRY FOR STUDENTS OF MEDICINE. Pp. 227. London: Longmans. Price 8s. 6d. net.

CURTIS TESTED SANDS FOR ALL INDUSTRIAL PURPOSES. (Trade Catalogue). Westmoor Laboratory, Chatteris.

Gives in alphabetical order the characteristics (physical and chemical) of 40 different kinds of sands used in various industries.

FEBRUARY, 1925.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS
AND OTHER ANALYTICAL CHEMISTS.

The Influence of Hydrogen Ion Concentration on the Colorimetric Determination of Pyrogallol and Catechol Derivatives

By S. GLASSTONE, M.Sc., Ph.D., F.I.C.

(*Read at the Meeting, December* 3, 1924.)

Mitchell (Analyst, 1923, 48, 2) has developed a colorimetric method for the quantitative estimation of pyrogallol tannins which depends on the production of a reddish-violet colour on the addition of a reagent containing ferrous sulphate and Rochelle salt to a very dilute solution of the tannin; the intensity of the colour produced appears to be the same for one equivalent of a pyrogallol nucleus independent of its actual state of combination. Mitchell has found this condition to hold good for gallic acid and gallotannin (tannic acid), and the author has been able to confirm his conclusions. Miss Price (*ibid.*, 1924, 49, 361) has attempted to extend the method to the estimation of catechol derivatives, but the results have been disappointing, perhaps, as she suggested, because of the tendency of the catechol nucleus to form abnormal complex iron salts. It will be seen below, however, that other factors are involved.

The work of Mitchell and of Miss Price has been criticised (Analyst, 1924, **49,** 168) on the grounds that the reaction should have been carried out at a more or less definite hydrogen ion concentration. When one considers the fact that the addition of acid converts the violet colour produced by mixing Mitchell's reagent with a pyrogallol or catechol derivative into a pale greenish-yellow colour, whilst the addition of alkali produces an orange colour, it will be realised that this criticism may have some foundation. It therefore appeared to be of some interest to investigate the limits of hydrogen ion concentration between which it is possible to obtain the violet colour described by Mitchell, and also to determine the conditions under which the maximum colour for any substance could be produced. From the results of the experimental work described below it is seen that different substances have different hydrogen ion limits for the formation of the violet colour, and that, unless precautions are taken to see that the conditions are such as to be well within the limits of hydrogen ion concentration for each substance, two solutions which contain equivalent amounts of pyrogallol or of catechol nucleus may not give the same intensity of colour. This appears to be the reason for the disappointing results obtained by Miss Price (*loc. cit.*), although it is shown below that her results are not so bad as would appear at first sight. It should be mentioned at the outset that the violet colour given by pyrogallol derivatives is much more intense than that obtained with catechol derivatives; the maximum colour intensities of equivalent solutions of pyrogallol and catechol have been found to be in the ratio of 2:1 (Miss Price, *loc. cit.*, p. 363, gives 8:3 as a result of direct comparison between pyrogallol and catechol, but from her figures for gallic acid and protocatechuic acid the calculated ratio is almost 2:1). It is, therefore, not quite satisfactory to compare derivatives of pyrogallol with those of catechol, and the substances in each series of derivatives should only be compared among themselves.

EXPERIMENTAL.—The object of these experiments was to determine the limits of hydrogen ion concentration between which it was possible to obtain the violet colour by adding the ferrous tartrate reagent to solutions of pyrogallol, gallic acid, tannic acid, catechol and protocatechuic acid. A small quantity (about 0·0002 grm.) of the phenolic derivative was mixed with 2 c.c. of the ferrous tartrate reagent in order to produce a distinct violet colour, and then 10 c.c. of a 10 per cent. solution of ammonium acetate were added to act as a "buffer" and so prevent any large and sudden changes in the hydrogen ion concentration. The violet-coloured solution was diluted and divided into two parts; to one part sulphuric acid was added until the violet colour was only just perceptible, and to the other sodium hydroxide solution until the mixture showed a slight orange tint. The hydrogen ion concentration of each solution was then determined by measuring the potential of a platinised platinum electrode placed in the solution, through which a stream of purified hydrogen was meanwhile made to bubble; the results have been expressed in terms of P_H. All experiments were carried out at room temperatures (about 12°–14° C.). It should be mentioned that, although phenolic bodies are known to interfere with the satisfactory working of the hydrogen electrode, yet in the solutions examined these bodies were present in such small amounts as to exert no appreciable influence on the electrode. The following results were obtained:—

Phenolic Substance.	P_H Limits.
Pyrogallol	6·5 to 10·3
Gallic acid	5·9 to 10·3
Tannic acid	4·1 to 11·1
Catechol	7·0 to 10·3
Protocatechuic acid	6·3 to 10·4

DISCUSSION.—The results show clearly that the latitude permissible in hydrogen ion concentration varies with the substance investigated. If the experiments were carried out under conditions of exact neutrality, that is at P_H 7, catechol would not give the violet colour, pyrogallol would give it slightly, but gallic and tannic acids, and probably protocatechuic acid also, would give it distinctly. Working in the ordinary way, the ferrous sulphate solution, and also that of the phenolic body, would be acid, the Rochelle salt should be slightly alkaline, and, if tap water were used for purposes of dilution, the resulting mixture would have a P_H of about 7 to 7·5. In the case of pyrogallol derivatives the figures given above, as well as the quantitative results obtained by Mitchell (*loc. cit.*), indicate that these conditions are quite suitable for the production of the maximum violet colour, and so it is found that, even without taking any special precautions, equivalent amounts of pyrogallol, gallic and tannic acid give equal intensities of colour in Mitchell's test. When working with catechol and protocatechuic acid, however, Miss Price did not obtain satisfactory results; this is no doubt due to the fact that catechol requires a larger P_H (about 8) before its maximum colour is produced, whilst protocatechuic acid will give its maximum colour under ordinary conditions, that is with a P_H of 7 to 7·5. It is clear, therefore, that unless special precautions are taken, protocatechuic acid will give a more intense colour than an equivalent amount of catechol when Mitchell's test is applied in the ordinary way. This is actually what Miss Price has found to be the case. It has been found, however, that if the P_H value is so adjusted that catechol gives its maximum colour, it is of the same intensity as that given under maximum conditions by an equivalent amount of protocatechuic acid, and so Mitchell's ferrous tartrate test, with suitable modifications, can also be used for the quantitative estimation of the catechol nucleus. Although Miss Price compared catechol with protocatechuic acid, and catechol with catechin, she did not make any direct comparison of the acid with catechin; such a comparison, however, can be made from Miss Price's results with interesting consequences. For equal intensities of colour

the ratio $\frac{\text{catechol}}{\text{protocatechuic acid}}$ found is approx. 94:100 (Miss Price, *loc. cit.*, p. 362);

and ,, ,, $\frac{\text{catechol}}{\text{catechin}}$,, ,, ,, 100:213 (p. 362);

therefore ,, ,, $\frac{\text{protocatechuic acid}}{\text{catechin}}$ is 100×100:213×94=1:2 (approx.).

The theoretical ratio for these two substances is $\frac{172}{344}=1:2$. The results show that under the conditions of Miss Price's experiments Mitchell's method was quantitative when comparing protocatechuic acid and catechin, although she did not realise this fact. It is fairly evident that at a P_H of 7 to 7·5 catechin produces its maximum colour, as also does protocatechuic acid, but, as catechol only gives its maximum at a larger P_H value, it would appear that, under the conditions of the original experiments, catechol could not be compared with the two derivatives.

We may assume, in general, that the colour changes which result from alterations in the hydrogen ion concentration are due to varying equilibria between three substances A, B and C, viz. $A \rightleftarrows B \rightleftarrows C$, where A is yellow in solution, B violet and C orange; decrease of hydrogen ion concentration causes the equilibria to be displaced towards the right. In each group of derivatives, *i.e.* of pyrogallol or catechol, equivalent amounts of the B form appear to give the same intensity of colour in solution, but the actual position of the equilibrium will not necessarily be the same for each substance at any given hydrogen ion concentration, nor will the colour intensities necessarily be the same for each group. It follows, therefore, that one group may not be comparable with another, and also that the substances in one group are only comparable with one another when the same equilibrium point has been reached, that is when the ratio of B to A is the same in each case; this condition may be realised by varying the hydrogen ion concentration of the mixture of phenolic body and ferrous tartrate reagent.

It has been found that the gradual addition of dilute alkali to the slightly acid mixture of phenolic body and Mitchell's reagent first produces a faint violet colour which becomes reddish-violet and soon reaches a maximum intensity; further addition of alkali causes no alteration in the colour until the orange tint becomes evident. It thus appears that in the reaction $A \rightleftarrows B$, above a certain P_H value, the equilibrium is displaced almost completely to the right, but that the change from B to C does not become appreciable until there is a much greater increase in the P_H value. It is clear, therefore, that comparisons between different solutions in Mitchell's test are best made under such conditions that each substance is producing its maximum intensity of colour, independent of the actual value of the hydrogen ion concentration in each case. With pyrogallol derivatives it would appear that the ordinary method of making up the test solutions and using tap water for purposes of dilution gives the conditions for maximum intensity of colour, but the present author would recommend the addition of about 5 to 10 c.c. of 10 per cent. ammonium acetate solution to the mixture of 1 c.c. of phenolic body and 2 c.c. of ferrous tartrate reagent before diluting to 100 c.c. for purposes of comparison. The ammonium acetate acts as a buffer, and the resulting solution will always have a P_H of about 7·6, provided, of course, that the reagents do not contain any considerable amount of acid or alkali. Apart from neutralising acidity, the ammonium acetate has been found to have no influence on the colour. In the case of catechol derivatives, although a P_H of 7·6 appears to be sufficient to produce a maximum colour with catechin and protocatechuic acid, it does not do so with catechol itself.* With that substance it has been found that the addition of a little alkali (dilute ammonia solution) to the ammonium acetate, so as to make the P_H about 8, will be sufficient to produce the maximum colour, and this solution may be used for catechol derivatives also. The colour is then somewhat more reddish than that obtained with pyrogallol derivatives.

* Since writing this paper the author has been able to examine, thanks to the kindness of Dr. M. Nierenstein, a number of catechol derivatives; the P_H limits for the formation of the violet or reddish-violet colour have been found to be as follows:

Iso-acacatechin from acacia catechu	6·5	to	8·9
Acacatechin from acacia catechu	6·35		11·4
Gambier catechin from cube gambier	6·3		12·0
Catechin from Chinese rhubarb .	6·3		11·9
Catechin from W. Africa cacao bean .	6·3		11·8

In accordance with the views expressed in this paper, it has been found that the addition of ammonium acetate alone is invariably sufficient to produce the maximum intensity of colour; in some cases, however, it is necessary to add a little alkali to produce the reddish tint which has been found to be most satisfactory for the comparison of catechol derivatives.

When dealing with a substance for which the conditions for the production of the maximum intensity of colour are not known, two methods are available for discovering these conditions; the first is to determine the P_H at which the violet colour is very faint, and to work at about one unit of P_H above this value; the second, which is much more suitable for actual practice, is to make up a series of mixtures of 1 c.c. of the solution of the phenolic body, 2 c.c. of the ferrous tartrate reagent and 5 c.c. of 10 per cent. ammonium acetate solution, and then to add different amounts of dilute ammonia (about 0·25 *N*) to each mixture. In this way the conditions for the production of the maximum colour can be readily determined. Frequently the addition of ammonia solution will not be necessary at all. If the addition of ammonium acetate alone should produce the orange colour, dilute acid must be added until it disappears and the maximum violet colour is obtained. Solutions made up in this way appear to be comparable with one another independent of the actual hydrogen ion concentration in each case.

CONCLUSION AND SUMMARY.—The limits of hydrogen ion concentration between which certain phenolic bodies yield a violet colour when treated with Mitchell's ferrous tartrate reagent have been determined; they have been found to vary from one substance to another even in the same group. The conditions under which the test may be made quantitative for the estimation of pyrogallol or catechol derivatives have been examined both theoretically and practically, and a modification of Mitchell's original method has been suggested for the determination of these phenolic bodies.

The author desires to express his thanks to Mr. C. A. Mitchell and to Mr. Alan H. Ware, for calling his attention to the problem dealt with in this paper

UNIVERSITY COLLEGE, EXETER.

The Determination of Coconut Oil and Butter Fat in Margarine.

BY G. D. ELSDON, B.SC., F.I.C., AND PERCY SMITH.

(*Read at the Meeting, October* 1, 1924.)

SOME time ago the authors were engaged in legal proceedings which turned on the amount of butter which was present in a so-called "Margarine blended with butter."

There was some difference of opinion among those engaged in the case as to the amount of butter present in the sample, and as the authors considered that there is a general tendency to over-estimate very small quantities of butter in margarine, and as this opinion was strengthened by the fact that when the formula of Bolton, Revis and Richmond (*vide infra*) was applied to the results obtained by them with their mixtures containing no butter (Table I.) small quantities of butter of the order of 0·5 per cent. were indicated, it was decided to reinvestigate the whole matter. This has been done and the results obtained are given below.

The Correction of Cribb and Richards.—Since the Polenske and Kirschner processes were first published many more or less useful suggestions have been made with regard to the interpretation of the results obtained by these processes, and the calculation of the amounts of butter, coconut oil and oleo-margarine present in the mixtures examined. Of these suggestions, the first really important one dealt with the Reichert and Polenske processes only, namely, that of Cribb and Richards (Analyst, 1911, 36, 327), whose work was extended by Arnaud and Hawley (Analyst, 1912, 37, 122). The main point of this work was that in the case of mixtures containing coconut oil and butter fat the Reichert and Polenske figures are not directly proportional to the amount of fat present, or, in other words, that the Reichert and Polenske values obtained by experiment ought to be subject to correction before the amounts of the fats could be calculated by direct proportion. The following formulæ were suggested for the calculation of the true Polenske and Reichert values:

$$P' = P - 0{\cdot}4 - R/10 + T,$$
$$R' = R - 0{\cdot}3 - RC - T,$$

where P′, R′, P and R are the true and observed Polenske and Reichert values, respectively, and RC is the Reichert value theoretically due to the coconut oil present, whilst T is a correction, which is 1·9 when the Polenske value is above 2·5 and becomes less when the Polenske value is below 2·5, in accordance with the curve given by Arnaud and Hawley. This correction is a very important point, and will be dealt with in detail later.

The above work mainly dealt with the determination of coconut oil, but Revis and Bolton (Analyst, 1911, 36, 333) and later these authors with Richmond (Analyst, 1912, 37, 183) also took up the work particularly in regard to the Kirschner process and the determination of small quantities of butter fat and it is largely to these authors, together with the later work of Cranfield, that we are indebted for our present knowledge of the value of the Kirschner process. As a result of a large amount of work they arrived at the formula

$$\text{Butter fat, per cent.} = \frac{K - (0{\cdot}262P^{0{\cdot}63} + 0{\cdot}09)}{0{\cdot}242},$$

which may be simplified with fairly accurate results, to

$$\text{Butter fat, per cent.} = \frac{K - (0{\cdot}1P + 0{\cdot}24)}{0{\cdot}244},$$

which means that the Kirschner figure is practically proportional to the amount of butter fat present.

These authors also dealt with the relation between the Kirschner and Polenske values for butter fat. This work has been continued by Cranfield (Analyst, 1915, 40, 439) and by Richmond (Analyst, 1919, 44, 166), who show that

$$P = 0{\cdot}26(K - 14).$$

The last author also gives the relationship between the Reichert-Meissl and Polenske values as follows:

$$0{\cdot}033R - 0{\cdot}6155 = \log 10(P - 0{\cdot}48).$$

In order to test the validity of the correction suggested by Cribb and Richards, the experimental figures given by Bolton, Revis and Richmond (*vide supra*) have been compared with the calculated values,* and the figures so obtained are set out in the following table:—

TABLE I.

		Percentage of Butter Fat.							
		0		2		5		10	
Coconut Oil. Per Cent.	Process.	Expt.	Calc.	Expt.	Calc.	Expt.	Calc.	Expt.	Calc.
0	Reichert	0·4	—	0·9	1·0	1·7	1·9	3·3	3·4
	Polenske	0·3	—	0·4	0·4	0·4	0·5	0·6	0·6
	Kirschner	0·2	—	0·8	0·7	1·5	1·4	2·7	2·6
5	Reichert	0·9	0·8	1·7	1·4	2·2	2·3	4·1	3·8
	Polenske	0·7	1·1	0·8	1·2	0·7	1·3	0·9	1·4
	Kirschner	0·2	0·3	0·7	0·8	1·3	1·5	2·9	2·7
10	Reichert	1·6	1·2	2·4	1·8	3·4	2·7	4·9	4·2
	Polenske	1·2	1·9	1·2	2·0	1·4	2·1	1·3	2·2
	Kirschner	0·3	0·4	0·9	0·9	1·6	1·6	3·0	2·8
15	Reichert	2·7	1·6	3·2	2·2	4·3	3·1	5·5	4·5
	Polenske	1·7	2·7	1·8	2·8	1·7	2·9	2·0	3·0
	Kirschner	0·4	0·5	0·9	1·0	1·7	1·7	3·0	2·9
25	Reichert	3·9	2·3	4·9	2·9	5·5	3·8	6·5	5·3
	Polenske	2·9	4·2	2·6	4·3	3·0	4·4	2·9	4·5
	Kirschner	0·5	0·7	1·2	1·2	1·9	1·9	3·1	3·1
50	Reichert	6·1	4·2	6·6	4·8	7·7	5·7	9·1	7·0
	Polenske	7·1	8·1	7·2	8·2	6·9	6·3	7·3	8·4
	Kirschner	1·1	1·1	1·6	1·6	2·3	2·3	3·4	3·5
75	Reichert	7·0	6·2	8·3	6·8	9·2	7·7	10·5	9·2
	Polenske	12·4	12·1	11·9	12·2	12·2	12·3	12·3	12·4
	Kirschner	1·5	1·5	1·9	2·0	2·6	2·7	3·7	3·9
100	Reichert	8·1	—	—	—	—	—	—	—
	Polenske	16·0	—	—	—	—	—	—	—
	Kirschner	2·0	—	—	—	—	—	—	—

On examining these figures it will be noticed that the 1·9 correction proposed by Cribb and Richards is only very approximately correct, and even so, only for proportions of coconut oil lying between 25 and 50 per cent. In order that this fact may be observed more clearly the differences between the observed and calculated values are set out in the following table:—

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* The theoretical values calculated, from the known constants of the constituents of the mixtures used, by simple proportion.

Table II.

Correction for Reichert and Polenske Values, B., R. & R.

Add to observed values.

	Reichert Value.					Polenske Value.			
Coconut oil.	Percentage of Butter.				Coconut oil.	Percentage of Butter.			
Per Cent.	0	2	5	10	Per Cent.	0	2	5	10
0	0·0	0·1	0·2	0·1	0	0·0	0·0	0·1	0·0
5	-0·1	-0·3	0·1	-0·3	5	0·4	0·4	0·6	0·5
10	-0·4	-0·6	-0·7	-0·7	10	0·7	0·8	0·7	0·9
15	-1·1	-1·0	-1·2	-1·0	15	1·0	1·0	1·2	1·0
25	-1·6	-2·0	-1·7	-1·2	25	1·3	1·7	1·4	1·6
50	-1·9	-1·8	-2·0	-2·1	50	1·0	1·0	1·4	1·1
75	-0·8	-1·5	-1·5	-1·3	75	-0·3	0·3	0·1	0·1

Some time ago the authors commenced a detailed study of the Blichfeldt process (*J. Soc. Chem. Ind.*, 1910, **29**, 792) and its modifications (*ibid.*, 1919, **38**, 150T; Analyst, 1920, **45**, 2) and for this purpose prepared a large number of mixtures of oleo-margarine, coconut oil and butter. Although, for various reasons which will be mentioned later, the study of the Blichfeldt process was not continued, the series of mixtures was examined by the Reichert-Polenske-Kirschner process. The figures obtained are given in Table III. opposite, together with the values calculated from the quantities of the various constituents present. The determinations described as "soluble" will be dealt with later. Each value has been determined in duplicate, and in every case where the first two determinations have not agreed they have been repeated until complete concordance has been obtained. In some cases as many as eight different determinations have been made on one value in order to remove all possibility of doubt as to the true figure, but in no case has the original disagreement exceeded 0·3 c.c. It follows, therefore, that, whatever value this work may have, at least no pains have been spared to make it as reliable as possible.

It is, perhaps, somewhat unfortunate that the margarine base for these mixtures had a somewhat higher Polenske value than usual, but this fact will scarcely influence the results which have been obtained, as will be seen from a study of the figures themselves.

The differences between the observed and the calculated values are tabulated below. It will be seen that, although they bear a general relationship to those obtained from the figures of Bolton, Revis and Richmond, yet the differences are not inconsiderable, and it is submitted that these latest figures show less variation among themselves than those of the others mentioned. As an example, one might point to the variations in the Reichert corrections obtained by Bolton, Revis and Richmond for mixtures containing 25 per cent. of coconut oil. With no butter,

TABLE III.

Coconut oil. Per Cent.	Process.	Percentage of Butter Fat.											
		0		2		4		6		8		10	
		Expt.	Calc.	Expt.	Calc.	Expt.	Calc.	Expt.	Calc.	Expt.	Calc.	Expt.	Calc.
0	Reichert	0·2	0·2	0·7	0·8	1·2	1·4	1·8	1·9	2·4	2·5	2·9	3·1
	Polenske	0·7	0·7	0·8	0·7	0·9	0·8	1·0	0·9	1·1	0·9	1·2	0·9
	Kirschner	0·2	0·2	0·7	0·7	1·2	1·2	1·7	1·7	2·2	2·2	2·8	2·7
	Soluble	0·2	0·2	0·7	0·7	0·8	1·2	1·4	1·7	1·9	2·2	2·2	2·7
5	Reichert	0·9	0·6	1·5	1·2	2·0	1·7	2·7	2·3	3·2	2·9	3·8	3·5
	Polenske	1·2	1·6	1·2	1·6	1·6	1·7	1·8	1·7	2·0	1·8	2·2	1·8
	Kirschner	0·3	0·3	0·7	0·8	1·2	1·3	1·7	1·8	2·2	2·3	2·6	2·8
	Soluble	0·1	0·2	0·4	0·8	1·0	1·3	1·7	1·8	2·3	2·3	2·9	2·8
10	Reichert	1·6	1·0	2·0	1·5	2·7	2·1	3·3	2·7	3·8	3·3	4·2	3·9
	Polenske	1·6	2·4	1·8	2·4	1·9	2·4	2·1	2·5	2·3	2·5	2·4	2·6
	Kirschner	0·4	0·4	0·8	0·9	1·3	1·4	1·9	1·9	2·4	2·5	2·9	2·9
	Soluble	0·5	0·3	0·8	0·8	1·5	1·3	2·2	1·8	2·9	2·3	3·3	2·8
15	Reichert	2·3	1·4	2·7	1·9	3·4	2·5	4·0	3·1	4·4	3·7	4·9	4·3
	Polenske	2·2	3·2	2·4	3·3	2·6	3·3	2·8	3·3	3·0	3·4	3·2	3·4
	Kirschner	0·5	0·5	0·9	0·9	1·4	1·5	1·9	2·0	2·5	2·5	3·1	3·0
	Soluble	0·8	0·4	1·1	0·9	1·9	1·4	2·6	1·9	3·1	2·4	3·7	2·9
20	Reichert	2·9	1·7	3·3	2·3	3·9	2·9	4·5	3·5	5·0	4·0	5·5	4·6
	Polenske	2·4	4·0	2·5	4·0	2·8	4·1	3·0	4·1	3·2	4·2	3·3	4·2
	Kirschner	0·6	0·5	1·0	1·0	1·5	1·5	2·0	2·0	2·7	2·5	3·3	3·1
	Soluble	1·1	0·4	1·6	0·9	2·2	1·4	2·9	1·9	3·6	2·4	4·2	3·0
30	Reichert	4·0	2·5	4·4	3·1	5·0	3·6	5·5	4·2	6·2	4·7	6·7	5·3
	Polenske	3·6	5·6	3·7	5·7	3·9	5·7	4·1	5·8	4·4	5·8	4·5	5·9
	Kirschner	0·8	0·7	1·3	1·2	1·8	1·7	2·3	2·2	2·8	2·7	3·3	3·2
	Soluble	1·2	0·5	1·8	1·0	2·4	1·5	3·1	2·0	3·9	2·5	4·2	3·0
40	Reichert	4·5	3·2	5·1	3·8	5·5	4·4	6·0	4·9	6·4	5·5	6·9	6·1
	Polenske	4·8	7·2	5·2	7·3	5·4	7·3	5·6	7·4	5·7	7·4	5·9	7·5
	Kirschner	1·0	0·8	1·5	1·3	2·0	1·8	2·5	2·3	3·1	2·8	3·5	3·3
	Soluble	1·2	0·6	1·8	1·1	2·5	1·6	3·2	2·1	4·0	2·6	4·7	3·1
50	Reichert	5·1	4·0	5·7	4·5	6·2	5·1	6·6	5·7	7·2	6·3	7·4	6·9
	Polenske	6·7	8·9	6·8	8·9	7·0	9·0	7·1	9·0	7·3	9·1	7·4	9·1
	Kirschner	1·2	1·0	1·5	1·5	2·0	2·0	2·5	2·5	3·1	3·0	3·7	3·5
	Soluble	1·3	0·7	2·1	1·2	2·6	1·7	3·0	2·2	3·7	2·7	3·9	3·2
60	Reichert	5·5	4·7	6·0	5·3	6·3	5·9	6·6	6·4	7·0	7·0	7·5	7·6
	Polenske	9·4	10·5	9·6	10·6	9·8	10·6	10·0	10·7	10·2	10·7	10·3	10·8
	Kirschner	1·3	1·1	1·7	1·6	2·2	2·1	2·7	2·6	3·3	3·1	3·7	3·6
	Soluble	1·1	0·8	1·6	1·3	2·0	1·8	2·3	2·3	2·8	2·8	3·4	3·3
70	Reichert	6·2	5·5	6·6	6·1	7·2	6·7	7·6	7·2	8·1	7·8	8·6	8·3
	Polenske	12·3	12·2	12·2	12·2	12·3	12·3	12·4	12·3	12·6	12·4	12·8	12·4
	Kirschner	1·3	1·2	1·7	1·7	2·2	2·2	2·7	2·7	3·2	3·2	3·8	3·7
	Soluble	1·4	0·9	1·8	1·4	2·5	1·9	2·9	2·4	3·5	2·9	3·9	3·4
80	Reichert	6·9	6·2	7·4	6·8	7·9	7·4	8·4	7·9	8·8	8·5	9·3	9·0
	Polenske	14·1	13·8	14·4	13·8	14·5	13·9	14·6	13·9	14·7	14·0	14·8	14·0
	Kirschner	1·4	1·4	1·8	1·9	2·3	2·4	2·9	2·9	3·4	3·4	3·8	3·9
	Soluble	1·4	1·0	1·9	1·5	2·5	2·0	3·0	2·5	3·5	3·0	4·1	3·5
90	Reichert	7·3	6·9	7·7	7·5	8·3	8·0	8·7	8·6	9·2	9·2	9·7	9·8
	Polenske	15·6	15·5	15·7	15·5	15·8	15·5	16·0	15·6	16·0	15·6	16·4	15·6
	Kirschner	1·5	1·5	2·1	2·0	2·6	2·5	2·8	3·0	3·2	3·5	3·7	4·1
	Soluble	1·4	1·1	1·9	1·6	2·5	2·1	3·1	2·6	3·6	3·1	4·3	3·7

Butter:—R = 29·0; P = 2·6; K = 24·8. Coconut oil:—R = 7·7; P = 17·1; K = 1·7; Soluble 1·2.

their correction is 1·6, with 2 per cent. of butter the correction is 2·0, with 5 per cent. of butter it falls to 1·7, and with 10 per cent. of butter it again falls to 1·2, whereas no such variations are observed in the case of the mixtures with 15 per cent. of coconut oil. In the case of the similar mixtures prepared by the authors no such fluctuations have been observed, and it would therefore appear that the figures based on these have a somewhat greater claim to accuracy than the former. It will be seen that the greatest corrections are required when the Polenske value varies between 2·5 and 9·0; Bolton, Revis and Richmond have only two mixtures which come within these limits, and even these are almost at the two extremes. It seems quite probable that had these workers made other mixtures with 30, 40 and 60 per cent. of coconut oil they might have obtained different results.

TABLE IV.

Corrections for Reichert and Polenske values, E. & S.

Add to observed values.

REICHERT VALUE.

Coconut oil. Per Cent.	Percentage of Butter. 0	2	4	6	8	10
0	—	+0·1	+0·2	+0·1	+0·1	+0·2
5	−0·3	−0·3	−0·3	−0·4	−0·3	−0·3
10	−0·6	−0·5	−0·6	−0·6	−0·5	−0·3
15	−0·9	−0·8	−0·9	−0·9	−0·7	−0·6
20	−1·2	−1·0	−1·0	−1·0	−1·0	−0·9
30	−1·5	−1·3	−1·4	−1·3	−1·5	−1·4
40	−1·3	−1·3	−1·1	−1·1	−0·9	−0·8
50	−1·1	−1·2	−1·1	−0·9	−0·9	−0·5
60	−0·8	−0·7	−0·4	−0·2	0·0	+0·1
70	−0·7	−0·5	−0·5	−0·4	−0·3	−0·3
80	−0·7	−0·6	−0·5	−0·5	−0·3	−0·3
90	−0·4	−0·2	−0·3	−0·1	0·0	+0·1

POLENSKE VALUE.

Coconut oil Per Cent.	Percentage of Butter 0	2	4	6	8	10
0	—	−0·1	−0·1	−0·1	−0·2	−0·3
5	+0·4	+0·4	+0·1	−0·1	−0·2	−0·4
10	0·8	0·6	0·5	0·4	0·2	0·2
15	1·0	0·9	0·7	0·5	0·4	0·2
20	1·6	1·5	1·3	1·1	1·0	0·9
30	2·0	2·0	1·8	1·7	1·4	1·4
40	2·4	2·1	1·9	1·8	1·7	1·6
50	2·2	2·1	2·0	1·9	1·8	1·7
60	1·1	1·0	0·8	0·7	0·5	0·5
70	−0·1	0·0	0·0	−0·1	−0·2	−0·4
80	−0·3	−0·6	−0·6	−0·7	−0·7	−0·8
90	−0·1	−0·2	−0·3	−0·4	−0·4	−0·8

It will be noticed that the total volatile acids (the sum of the Reichert and Polenske values) do not vary much from the calculated values. This will be seen by adding together the Polenske and Reichert values and comparing them with the sum of the calculated values. It follows, therefore, that the variations observed are due, not to any great variation in the total amount of volatile acids distilled, but to the varying solubility of the Polenske acids in the acids contained in the Reichert distillate, as we are not concerned with the solubility of acids in water but their solubility in an aqueous solution of other acids.

CALCULATION OF AMOUNT OF COCONUT OIL.—The calculation of the percentage of coconut oil from the observed Reichert and Polenske values is, therefore complicated by the fact that these values are not strictly proportional to the amount of coconut oil present, but it is submitted that if a suitable correction be taken from Table IV., on the lines first suggested by Cribb and Richards, the corrected Reichert and Polenske values then become proportional to the amount of butter and coconut oil present, and that the calculation of the amount is merely based on proportion and does not involve any complicated formula. It will be seen that the correction varies not only with the amount of coconut oil present but also with the amount of butter, so that in determining the amount of coconut oil present in a mixture by the Polenske process it is first necessary to determine the amount of butter by the Kirschner process.

As it is now most unusual for mixtures of fat to contain more than 10 per cent. of butter fat, it has not been considered worth while to continue these mixtures above 10 per cent. of butter. The authors agree with Bolton, Revis and Richmond that the Kirschner value is directly proportional to the amount of butter present; the calculated and observed values of this figure nearly always agreeing to 0·1 which is not outside the possible experimental error. The percentage of butter fat may be calculated (as a first approximation) from the Kirschner value in the following way:—

$$\text{Per cent. butter fat} = \frac{K - 0{\cdot}2 - P/10}{23{\cdot}5},$$

where K is the observed Kirschner value, P is the observed Polenske value, and 23·5 the average Kirschner value for butter fat. It will be seen from a study of Table III. that the term P/10 is not accurate, and that it should vary both with the Polenske value and with the Kirschner value. To give these two points their full mathematical significance would, however, be to attempt to carry the interpretation of the results beyond the experimental accuracy of the process, and the following simple equations will give the percentage of butter fat with a near approach to accuracy.

POLENSKE VALUES.	EQUATION.
Less than 2	$\frac{K-0{\cdot}3}{0{\cdot}235}$
2·0— 4·5	$\frac{K-P/6-0{\cdot}2}{0{\cdot}235}$
5·0— 7·0	$\frac{K-P/6-0{\cdot}1}{0{\cdot}235}$
7·0— 9·0	$\frac{K-P/7-0{\cdot}1}{0{\cdot}235}$
9·0—10·0	$\frac{K-P/8-0{\cdot}1}{0{\cdot}235}$
10·0—12·0	$\frac{K-P/10-0{\cdot}1}{0{\cdot}235}$
12·0—17·0	$\frac{K-P/10}{0{\cdot}235}$

TABLE V.

Corrections for Observed Reichert and Polenske Values.

POLENSKE VALUES.

Observed value.	Correction to be added. Percentage of butter. 0	5	10
1·0	0·4	−0·1	−0·2
1·5	0·7	0·0	−0·2
2·0	0·9	0·5	−0·2
2·5	1·5	0·6	0·2
3·5	2·0	1·5	0·9
5·0	2·4	1·8	1·5
7·0	2·2	2·0	1·7
8·0	1·7	1·6	1·3
9·0	1·2	1·2	0·9
10·5	0·5	0·7	0·5
12·0	0·0	0·0	−0·3
14·0	−0·2	−0·6	−0·6
16·0	−0·1	−0·3	−0·7

REICHERT VALUES.

* Coconut oil. Per Cent.	Correction to be subtracted. Percentage of butter. 0	5	10
0	0·0	0·1	0·2
5	0·3	0·3	0·3
10	0·6	0·5	0·3
15	0·9	0·9	0·6
20	1·2	1·0	0·9
30	1·5	1·4	1·4
40	1·3	1·1	0·8
50	1·1	1·0	0·5
60	0·6	0·4	0·0
70	0·7	0·5	0·3
80	0·7	0·5	0·3
90	0·4	0·2	0·0
100	0·0	—	—

* Calculated from corrected Polenske value.

The amount of coconut oil may be calculated from the corrected Polenske value as follows:—

$$\text{Coconut oil, per cent.} = \frac{(P' - 0{\cdot}2 - 0{\cdot}03B) \times 100}{17{\cdot}6}.$$

where P′ is the corrected Polenske value, 17·6 is the average Polenske value of coconut oil, and B the percentage of butter deduced from the Kirschner value. The value 0·2 which is subtracted is to allow for the Polenske value of the non-coconut base; this figure should, of course, alter with the amount of the base present, being slightly lower in the case of a higher Polenske and slightly higher, say 0·3, where the Polenske value is lower, showing little coconut oil to be present.

The amount of coconut oil present having been thus calculated, the correction for the Reichert value may be found from the table, and the corrected figure then used to determine the percentage of butter fat in the following way:—

$$\text{Butter fat, per cent.} = \frac{(R' - 0{\cdot}065C - 0{\cdot}2) \times 100}{28{\cdot}4}.$$

where R′ is the corrected Reichert value, C is the percentage of coconut oil calculated from the previous equation, and 28·4 is the average Reichert figure for butter fat. This figure can then be compared with the amount of butter calculated from the Kirschner value.

These corrections will possibly not be applicable to mixtures containing palm-kernel oil and other oils of the same family. The authors are at present carrying out experiments along these lines and hope to publish their results in the immediate future. The matter, of course, has already been dealt with by Bolton, Revis and Richmond (*vide supra*), but the figures which they give require extending. It would seem advisable that, in all cases where very small quantities of butter fat are being looked for, mixtures should be made which give identical figures in the same apparatus as the sample in question. By this method most of the complicated factors will be cut out. It is hoped that the present work and that about to be published will give some assistance in the preparation of such mixtures.

THE BLICHFELDT PROCESS.—This process was first described in 1910 (*J. Soc. Chem. Ind.*, 1910, **29**, 792); further details were published in 1919 (*ibid*, 1919, **38**, 150T), and a modification has been described by Gilmour (ANALYST, 1920, **45**, 2). The authors have examined the original process of Blichfeldt, but they do not consider that it holds out sufficient advantages over the Reichert-Polenske-Kirschner process to justify the giving up of the latter which is now recognised as a standard process.

The method of Gilmour is ingenious and certainly, at first sight, appears to contain novel and important suggestions; it is hoped to discuss this process in full at a later date, as a modified method is now being worked out in this laboratory.

The writers have tried some experiments in the form of a modification of the Blichfeldt process. The Blichfeldt process deals with the whole of the volatile acids. It appeared possible that it might be useful to separate by means of the silver salts, the soluble and insoluble volatile acids separately, in place of dealing with them together, as is done in the Blichfeldt process. This was tried, but it was found that practically the whole of the silver salts of the Polenske fatty acids were insoluble in water, and that no useful purpose would be served by attempting their separation. The separation of the silver salts of the Reichert acids, however, is practically the Kirschner process and, as the method of Blichfeldt does not require the second distillation, it was thought that it might be a greater convenience to follow the procedure of Blichfeldt rather than that of Kirschner. This was done in the following method:—

Thirty-five c.c. of 0·1 *N* silver nitrate solution are added to the neutralised Reichert distillate, the whole transferred to a 220 c.c. flask, diluted to the mark with water and allowed to stand for one hour. After this time 200 c.c. are filtered off and titrated with 0·1 *N* ammonium thiocyanate, with iron alum as indicator. To the number of c.c. used in the titration one-tenth is added and also the value obtained in a blank experiment (usually about 0·2), and this value is subtracted from the original 35 c.c. This figure is then subjected to a further correction of one-tenth, so that it may be compared directly with the Reichert value. This final figure represents the amount of acids present of which the silver salts are insoluble under the conditions thus obtained; the soluble figure is found by subtracting this from the Reichert value. The value so obtained is the value referred to as "Soluble" in Table III.

It will be seen at once that the figures are not as good as those obtained by the Kirschner process. For small additions of butter they are not quite so responsive, and, further, there are greater differences between the observed and calculated figures than those shown by the Kirschner process. This modification of the Blichfeldt process may, however, be useful to those who object to the second distillation of the Kirschner process; it has the advantage over the Blichfeldt process that the figure can be obtained as an extension of the Reichert-Polenske process, and also there is, of course, a considerable saving in time. A somewhat similar method has been published by van der Laan since this work was done, but his results are apparently not so good as those obtained above (*cf. J. Soc. Chem. Ind.*, 1923, **42**, 287A).

DISCUSSION.

Mr. A. MORE remarked that the author had started off to prepare certain mixtures, but did not give any evidence of the extent of variation in the results which would be obtained from mixtures of coconut oil, palm-kernel oil, neutral fat, and butter fat, which contained the same proportions of the constituents, but differed in the analytical constituents of these constituents. There appeared to be no formula that would cover all possible mixtures. In the application of a formula one could not assume that the mixture being analysed contained coconut fat, and a worker must be prepared to meet possibilities. It was true that, in the case of certain mixtures, Mr. Elsdon had made out that the Government Laboratory figures were abnormal. Various formulæ for determining butter had been devised from time to time. The Reichert or Kirschner values of the constituents, especially of the butter fat, were bound to have a great influence on the results, and in calculating the quantity of butter fat in a mixture by means of a formula devised for average fats it was necessary to consider the range of the results due to the use of butters of varying composition. "One or two per cent. depending on the butter fat used in the mixture" was the form adopted in his certificate to indicate this variation. On the face of it Mr. Elsdon's process seemed elaborate.

Dr. BERNARD DYER said that he considered that a discussion as to whether there was 1 per cent. or 2 per cent. of butter in margarine was of little moment to the man in the street, though it might be of interest as a legal disputation.

Mr. G. D. ELSDON, referring to the number of samples, said that, of the data previously worked out, there were no samples of the section between 25 and 50 per cent., and that it was between those figures that the greatest correction was required. In the 30 to 40 per cent. section the correction was large, and if only for that reason, the extended table would be useful. They all realised, of course, that there was no such thing as a standard butter; one must assume average figures. If one was going to assume the presence of between 1 and 2 per cent. of butter fat in a mixture, one of these figures was a long way from an average. Taking an average Kirschner value of 24 for the 2 per cent. figure, the 1 per cent. figure would require a Kirscher value of 12, which would be almost impossibly low.

The Fat of Goats' Butter.

BY H. DROOP RICHMOND, F.I.C.

IN their note published in the ANALYST (1924, **49**, 509), Messrs. Knowles and Urquhart give a few results of the composition of goats' butter, and on this base arguments, first showing that the sale of goats' butter as "butter" is possible, and second, that by the use of accepted methods, and especially by the use of formulæ given in my *Dairy Chemistry*, goats' butter might be condemned as adulterated with coconut oil.

The figures given by these authors for the percentage of coconut oil, as calculated by the formula given by me, are hopelessly wrong, and are due to misquoting the formula as I give it on p. 249 of *Dairy Chemistry* (*cf.* ANALYST, 1919, **44**, 167). I wrote the formulæ as

$$\text{“}C = \frac{P-P'}{14\cdot4} \times 100.$$

C = percentage of coconut oil.
P = Polenske figure.
P′ = mean Polenske figure from the table (calculated for a figure) equal to the Reichert-Wollny figure found + half the Polenske figure found.”

The authors leave out the words in brackets, materially altering the sense.

An illustration with their No. 1 sample will show the difference.

R.-W.=27·66; Polenske 8·65.

I calculate thus P′=mean Polenske figure for $(27\cdot66+\frac{8\cdot65}{2}=)31\cdot98=3\cdot2$,

$$\frac{8\cdot65-3\cdot2}{14\cdot4} \times 100 = 37\cdot8.$$

They have evidently calculated

$$P'=\text{mean Polenske figure for } 27\cdot66=2\cdot52+\frac{8\cdot65}{2}=6\cdot84,$$

$$\frac{8\cdot65-6\cdot84}{14\cdot4} \times 100 = 12\cdot65.$$

In the table below I give in the first column the actual apparent percentage of coconut oil given by my formula $C=\frac{P-P'}{14\cdot4}\times100$. Second, the Reichert-Wollny figure which would be given by this percentage of coconut oil, third, the remainder of the Reichert-Wollny figure which is due to the butter present, and fourth, the calculated Reichert-Wollny figure of the original butters.

	Per Cent. Coconut oil.	R.-W. value of this.	R.-W. value due to butter.	R.-W. value of the original butter.
1.	37·8	3·0	24·66	39·5
2.	33·6	2·7	22·38	33·7
3.	31·8	2·54	25·23	37·0
4.	27·7	2·22	25·00	34·6
5.	40·2	3·2	22·76	37·8
6.	20·3	1·62	22·85	28·6
7.	33·2	2·66	23·41	35·0
8.	15·1	1·21	25·68	30·3
9.	36·5	2·92	23·95	37·7

It is seen that, in the case of seven out of nine samples, the calculated Reichert-Wollny figures are so absurdly high as to indicate to the analyst that he is not dealing with an ordinary mixture of butter and coconut oil, but with some abnormal product to which the formula is not applicable. The iodine values calculated for the original butters in a similar manner are very high in relation to the Reichert-Wollny figures calculated. Further, if the analyst turns to the table given by Messrs. Knowles and Urquhart on page 511, and compares the Reichert-Wollny and Kirschner values, it will be seen that the Kirschner figures are extraordinarily low for the Reichert-Wollny figures for butter.

It is to be regretted that the authors of this note have devoted so much of their paper to drawing attention to the fact that goats' butter might be condemned as containing coconut oil if sold as butter, and so little to the sufficiently marked difference between genuine goats' butter and adulterated cows' butter which might form the basis of a method of distinguishing them.

I may add that I wrote my formulæ in *Dairy Chemistry* with the full knowledge that they would not apply to the fat of mammals other than the cows, including goats' butter, and I have no reason to think that this very rare article of commerce need cause any great modification of existing methods of detecting adulteration. As Messrs. Knowles and Urquhart have quoted so much from my *Dairy Chemistry*, I would like to quote from p. 1: "The expressions 'milk,' 'butter,' etc., must be taken as applying to the products derived from the cow, unless described to the contrary,"

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

ANALYTICAL NOTES ON TWO SAMPLES OF OLD BUTTER.*

Two samples of tinned butter, at least twenty years old, which had been kept continuously in the offices of the Dairy Commissioner, Ottawa, exposed summer and winter to room temperatures, have recently been analysed by the Division of Chemistry of the Dominion Experimental Farms.

Examination on September 16th showed that both tins were in good condition, hermetically sealed and tight. The excellent condition of the labels indicated that the tins had not been exposed to damp air. There was no evidence of "swells" or other damage to the containers.

No. 1, labelled "Pierre de Bacourt, Dorchester Que. Extra Finest Canadian Butter," had been packed dry in a round tin. On opening the tin the butter was found to be soft with no texture; in appearance it was oleaginous and curdled and of a light pale yellow colour. The odour was slightly rancid, but largely yeasty and not very unpleasant. The taste was very disagreeable, and the butter was non-edible.

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* *Cf.* ANALYST, 1907, 32, 80.

No. 2, labelled "Pure Canadian Butter" (Canadian Government Dairy Station, Calgary, Alta.) had been packed in brine in a rectangular tin, in two 1 pound portions, each wrapped in parchment paper. There was no liquid on opening the tin; the brine, as such, had disappeared. The butter possessed a very strong rancid odour. It was very soft, with a curdled appearance and had no texture. The colour was pale yellow with white spots or patches. It was absolutely non-edible.

ANALYSIS OF BUTTERS.

	No. 1. Per Cent.	No. 2. Per Cent.
Water	14·87	33·60
Fat	79·73	57·24
Curd	1·42	3·08
Ash*	3·96	6·11
	99·98	100·03
* Including salt ..	3·80	5·94

ANALYSIS OF BUTTER FATS.

	No. 1.	No. 2.
Butyro-refractometer reading at 25° C. ..	48·98°	43·44°
Iodine value	30·70	31·62
Reichert-Meissl value	31·10	25·45
Polenske value	2·03	2·75
Acid value	45·32	121·3
Percentage of free acid as oleic	22·45	60·08
Percentage of soluble fatty acids as butyric ..	5·25	4·11
Insoluble fatty acids	84·93	86·92
Saponification value	227·6	225·8

It will be seen that the refractometer readings are in each case decidedly lower than would be obtained with normal butters; this is particularly true of Butter No. 2 which had been packed in brine. The lowering of refractive indices is explained by the abnormal content of free fatty acids.

The iodine numbers are normal. The lower Reichert-Meissl value and the higher Polenske value for the fat of No. 2 would seem to indicate the conversion in this sample of the soluble volatile acids into insoluble volatile acids, due to the presence of a large amount of water. This deduction is further supported by the figures obtained for soluble and insoluble fatty acids.

The acid value in both samples is very high, especially that of No. 2, indicating the effect of hydrolysis of the glycerides consequent upon the large amount of water present. The saponification values are normal.

FRANK T. SHUTT.

DOMINION OF CANADA,
DEPARTMENT OF AGRICULTURE,
DIVISION OF CHEMISTRY.

LOSS OF STRENGTH OF SPIRIT KEPT IN OPEN BOTTLES.

In 1916 the defence that deficiency of spirit was due to "the bottle having been left open" was raised in cases when proceedings were taken for selling potable spirit below standard strength (not exceeding 25 degrees under proof according to the legislation of the Union of South Africa).

During the summers of 1916–17 and 1917–18 opportunity was taken to test the point. During the time of the observations the prevailing temperature of the laboratory frequently rose to 33° C. in the daytime, and at night it seldom fell below 8° C.

In the first place two ordinary spirit bottles (about 27 fl. ozs.) were filled to the extent of about four-fifths with spirit 29·3 degrees under proof. One bottle was left uncorked, and the other was corked with a used cork inserted just as it would be if the bottle were in use. The bottles were kept in a cupboard and the spirits were tested after 11, 29 and 67 days. It was found that the spirit in the corked bottle had lost nothing in alcoholic strength when tested after 67 days, in spite of the bottle having been opened and the strength of the spirit determined (by specific gravity) on the 11th and 29th days. The spirit in the open bottle was 30·7 degrees under proof on the 11th day, 31·7 degrees under proof on the 29th day, and 34·05 degrees under proof after 67 days. The loss from the open bottle was, therefore, 4·75 degrees of proof in 67 days, or one degree of proof in about 14 days, or about 0·07 degree of proof per day.

At the beginning of the summer of 1917–18 four ordinary spirit bottles were filled to the extent of about four-fifths with spirit (brandy) 23·6 degrees under proof. Two of the bottles were left open and two were corked with used corks. An open bottle and a corked bottle were placed on a shelf on the north wall (exposed to the sun) of the laboratory and an open bottle and a corked bottle were placed on a shelf on the south wall (shaded from the sun) of the laboratory. All the bottles were shaded from direct sunlight. Specific gravity determinations of the spirits were made at intervals of 2 or 3 days for a total period of 93 days (30th October, 1917, to 31st January, 1918). On each occasion only such quantity of spirit was taken from the bottle as was required for the determination and the quantity was returned to the bottle immediately. No loss of strength was suffered by the spirits in the corked bottles in spite of their having been opened 38 times before the final determination on the 93rd day. The following table shows the progressive loss of strength of the spirits in the open bottles, the prevailing temperatures being of the order already stated; the figures represent degrees under proof:

Day.	North Wall.	South Wall.	Day.	North Wall.	South Wall.	Day.	North Wall.	South Wall.
0	23·6	23·6	34	27·3	26·6	64	28·9	27·9
4	24·3	24·1	36	27·4	26·6	66	29·1	28·2
6	24·9	24·4	38	27·6	26·7	69	29·25	28·2
8	25·0	24·6	41	27·7	26·85	71	29·35	28·25
10	25·25	24·9	43	27·8	26·95	73	29·6	28·45
13	25·5	25·2	45	27·9	26·95	76	29·8	28·6
15	25·7	25·4	48	28·0	27·1	78	29·9	28·7
17	25·8	25·5	50	28·2	27·3	80	30·25	28·8
20	25·9	25·6	52	28·25	27·4	83	30·45	28·8
22	26·0	25·8	55	28·45	27·5	85	30·7	28·9
24	26·2	25·9	57	28·6	27·6	87	31·05	29·1
27	26·5	26·1	59	28·6	27·7	90	31·25	29·35
29	26·95	26·4	62	28·8	27·8	93	31·6	29·45
31	27·1	26·5						

The results show that the spirit kept in the open bottle at the warmer (north wall) of the two places lost 8 degrees of proof in 93 days which is equivalent to a loss of one degree of proof in between 11 and 12 days, or to a loss of about 0·09 degree of proof per day. The spirit kept at the cooler (south wall) place lost 5·85 degrees of proof in 93 days, which is equivalent to a loss of one degree of proof in about 16 days, or to a loss of about 0·06 degree of proof per day.

In spite of the higher prevailing temperature at which the observations were made it is noteworthy that the losses sustained were appreciably lower than those recorded by H. Lowe (ANALYST, 1924, 49, 135). The rather higher loss (0·15 degree of proof per day) found by Lowe is probably to be attributed to stronger diffusion by air convection current over the surface of the spirit in spite of the loose cardboard cover, and, in a measure, to the greater surface exposed in a flask than in a bottle.

J. McCRAE.
J. HAWKEN.

GOVERNMENT CHEMICAL LABORATORIES,
JOHANNESBURG, TRANSVAAL.

PREPARATION OF NESSLER'S SOLUTION.

THE sensitiveness of Nessler's solution varies considerably, and usually it increases with age. By the use of the following directions a solution which is very sensitive immediately after preparation can be made.

Dissolve 17·5 grms. of potassium iodide in 100 c.c. of water; next dissolve 15 grms. (a slight excess) of mercuric chloride in 300 c.c. of water, and mix the two solutions. Wash thoroughly by decantation the heavy precipitate that forms, and dissolve it in a solution of 17·5 grms. of potassium iodide in 100 c.c. of water, add a few drops of mercuric chloride solution till a red precipitate, insoluble on shaking, is produced, and dilute to about 500 c.c. Cool the beaker in ice water, and mix the liquid with so much of a 50 per cent. sodium hydroxide solution (previously diluted with 200 c.c. of water and cooled in ice water) as is equal to 105 grms. of sodium hydroxide. Cool well during mixing, and make up to 1 litre. The solution is left to settle and the clear portion decanted for use.

H. DROOP RICHMOND.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

REPORT OF THE CITY ANALYST FOR THE THIRD QUARTER, 1924.

OF the 1296 samples submitted during the quarter, 1113 were analysed under the Sale of Food and Drugs Acts, 1011 being bought informally (38 adulterated), and 102 being bought under the provisions of the Act (4 adulterated).

MILK. The total number of samples examined was 556, of which 454 were informal samples. Thirty-four of the informal and 4 of the formal samples were adulterated.

CREAM.—Three of 15 informal samples of cream contained boric acid (0·21 to 0·32 per cent.) without a declaratory label; the vendors were cautioned.

Four of the 19 informal samples of preserved cream did not comply with the regulations. One sample, in addition to the declaratory label, was marked "Thick Rich Cream—and Devonshire Clotted Cream Daily." The "and" was very small, so that the statement about Devonshire cream, apparently referred to the contents of the receptacle. Such a label may be legal, but it is certainly misleading.

CHEESE.—Two sample of *cream cheese* contained 70·6 and 72·5 per cent. of fat respectively; about 95 per cent. of the dry solids was fat. A sample of *milk cheese* contained 17·8 per cent. of fat (about 49 per cent. of the dry solids).

FLOUR.—Four of the 28 samples of flour and 4 of the 10 samples of self-raising flour contained persulphate or peroxide. Pending the report of the Government Committee, they were passed as genuine.

JAM.—A complaint that a bramble and apple jam contained an excess of seeds was found to be due to an accumulation of a large proportion of the seeds in the upper part of the jam.

ZINC OINTMENT.—One of 4 informal samples, while containing the right proportion of zinc oxide, had not been prepared with benzoated lard, although marked "B.P."

HEALTH DEPARTMENT.—Of the 62 samples examined, 28 were milk. One of the 16 samples taken under the Rag Flock Act contained 80 parts of soluble chlorine per 100,000 (limit 30 parts); it contained wood shavings, straw and grass. The vendor was cautioned.

J. F. LIVERSEEGE.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

FALSE TRADE DESCRIPTION.

ALLARD *v.* SELFRIDGE AND CO., LTD.

AN appeal was heard in the High Court on November 10th, 1924, from a decision of Mr. Mead, the Marlborough Street Magistrate, who had dismissed an information against Messrs. Selfridge and Co. for selling goods to which a false trade description had been applied. The facts, which were not in dispute, were that the respondents had sold as silk some stockings which were made of artificial silk. Their buyer had bought these from a merchant in a small way of business, and had not applied any tests which would have shown that they were not silk. The Magistrate had held that the buyer had acted innocently and in good faith, and that the defendants, who were responsible for their buyer, had the benefit of her innocence, this being, in his view, in accordance with the finding in *Christie, Manson and Woods* v. *Cooper* (1900; 2 Q.B. 522).

Section 2 (2) of the Merchandise Marks Act, 1887, provides that:—

> Every person who sells . . . any goods . . . to which any false trade description is applied . . . shall, unless he proves—
>
> (*a*) that, having taken all reasonable precautions against committing an offence against this Act, he had at the time of the commission of the alleged offence no reason to suspect the genuineness of the . . . trade description; and
>
> (*b*) that on demand made by or on behalf of the prosecutor, he gave all the information in his power with respect to the persons from whom he obtained such goods or things; or
>
> (*c*) that otherwise he had acted innocently;
>
> be guilty of an offence against this Act.

Sir Duncan Kerly, K.C., for the appellant, said that, putting his argument in algebraical form, it should be a defence to prove either $a+b$ or c, but that the defendant could not prove $a+b-x$ and say that that was "c." In his contention c was intended to cover pure accident, such as the seller affixing a wrong lable or putting his hand into the wrong box. He submitted that the Magistrate was wrong in his decision for the following reasons:—(1) He had put the onus in the wrong place, deciding that the prosecution must show an intent to defraud; (2) he had thought that c must be complied with when there was no intent to defraud; (3) he had thought that even if the defence fall short of $a+b$, it was possible to prove c in a case to which $a+b$ were applicable and were relied upon for the defence; (4) because the Act threw on the seller the onus of giving a true description subject only to the defences of the section.

Mr. Hawke, K.C., for the respondent, contended that it was an answer to a charge under this section for the defendant to prove that he acted innocently, otherwise than by proving $a+b$. The Magistrate, in holding that the buyer had acted innocently, had found that there was no *mens rea*.

The Lord Chief Justice, in giving judgment, said that the Magistrate had decided the case upon the view that *mens rea* for the purpose of this statute meant an intent to defraud. The scheme of the Act was that once the forbidden act was proved, the offence was complete unless the accused proved one of a number of defences. These were of a twofold nature. He might prove (*a*) and (*b*) which were joined by the conjunction "and," or he might prove (*c*), and the words of (*c*) were not "that he has acted innocently," but "that otherwise he has acted innocently." The dichotomy was complete. But if, in seeking to prove (*a*) and (*b*) he failed to prove them apart, he could not have recourse to (*c*); to hold otherwise would involve two consequences, which were absurd—(1) that (*a*) and (*b*) were inserted merely as an illustration; (2) that the word "otherwise" must be omitted. In other words, if a seller sought to excuse himself on the ground that he had no reason to suspect the genuineness of the trade description, he must base that defence on having taken all reasonable precautions. What was done was an attempt to prove that not having taken all reasonable precautions, the respondents had no reason to suspect the genuineness of the trade description, which seemed to be nonsense.

The judgments in the case of *Christie, Manson and Woods* v. *Cooper* ought to be read strictly in conjunction with the facts of that case, and not extended. There the sellers were not putting forward an untrue trade mark as a true mark; although they could not erase it physically, they were doing their utmost to make it of no effect. A comparison of the decision in that case with that in *Stone* v. *Burn* (1911; 1 K.B., 927) seemed to make clear the reasons for the decision.

In the present case the magistrate seemed to have fallen into two errors. Apparently he had overlooked the word "otherwise" in the Sections 2 (2), and he seemed to have thought that a defence could be established by proving a part of what came under (*a*). Secondly, he seemed to have thought that *mens rea* for the purpose of this statute meant an intention to cheat and defraud. It meant an intention to infringe the Act of Parliament. It was not necessary to inquire into what was meant by (*c*). Where the words "acted innocently" were qualified by the word "otherwise" a defence could not be established by proving a portion of (*a*). The appeal must be allowed, and the case remitted to the Magistrate to convict.

Mr. Justice Shearman and Mr. Justice Salter gave judgments to the same effect.

METHYLATED SPIRIT AS CHERRY BRANDY.

On November 29th a Stepney tradesman, described as a watchmaker and mineral water manufacturer, was charged at the instance of the Board of Customs and Excise at Old Street Police Court with having prepared beverages named "Kummel" and "Cherry brandy" from methylated spirit, and also, not being an authorised methylator, with being in possession of methylated spirits obtained from a person not authorised to supply them. The firm that supplied him was also summoned for selling methylated spirits without authority or license, and the wholesale retailers were summoned for selling methylated spirits other than mineralised methylated spirits.

Counsel for the firm of licensed methylators said that they had received orders for "methylated finish," and that for some unknown reason pure spirit had been supplied. The firm was fined £40 with eight guineas costs, and one of its directors was fined £20 with four guineas costs for aiding and abetting in the sale.

The tradesman, on whose premises bottles of "Cherry brandy" and "Kummel," made up for the holidays, had been found, was fined £160 with nine guineas costs, and the Magistrate (Mr. Clarke Hall) said that he was sorry he had no power to recommend him for deportation.

IMPEDING AN INSPECTOR.

On December 31 a pharmacist was summoned, at Old Street Police Court, by the Bethnal Green Borough Council for having obstructed one of their inspectors under the Food and Drugs Acts in the course of his duties, and also for impeding the inspector in the course of his duties under the Acts.

Evidence was given that the inspector had, through his agent, obtained a bottle of medicine to be tested as to the accuracy of the dispensing of the prescription. This had been divided in the usual way, and no complaint as to the sample was alleged. On leaving the dispensary, the inspector went to another shop, giving his agent a prescription on the way. The defendant then entered the same shop and, according to the evidence of the inspector's agent, there was a whispered conversation between the two pharmacists. The inspector spoke to the defendant as he left the shop and said that, in his opinion, he had gone there with the object of giving a warning, and that he should report the matter to the medical officer and to the Insurance Commissioners.

Mr. Glyn Jones, for the defence, contended that a warning was not an obstruction or an impediment, and submitted that intentional misconduct, of which there was no evidence, was essential for the offence.

The Magistrate (Mr. Clarke Hall) considered that the defendant's action was both impeding and obstructing, within the meaning of Act. It was the inspector's duty to take samples, and he had gone to the shop to take them under such circumstances as those under which an ordinary customer would be supplied. Any action on the part of another person that would prevent that was, in his opinion, impeding the inspector in the course of his duties. Although he had no doubt that he was right, he was prepared to state a case, if required. If he were wrong, all the good, not only of the Food and Drugs Acts, but also of other Acts, would be destroyed. He would not inflict any penalty.

The summons for obstruction was withdrawn, and the defendant was ordered to pay five guineas costs on the other charge—impeding the inspector.

MIXING OF SAMPLES.

ON December 3rd a shop-keeper was summoned at Woolwich for having sold camphorated oil which contained mineral oil and was deficient in camphor. Evidence was given by an agent of the inspector that she had purchased two bottles of the preparation and had handed them to the inspector. The inspector said that, as the bottles were small, he had mixed their contents for the purpose of sampling.

The solicitor for the defence contended that the contents of the two bottles, which were sent out by different manufacturers and were differently described, should not have been mixed.

The Magistrate (Mr. Ratcliffe Cousins) agreed that this should not have been done, and dismissed the case with 2 guineas costs against the Borough Council.

Department of Scientific and Industrial Research.

REPORT OF THE FOOD INVESTIGATION BOARD FOR THE YEAR 1923.*

THE most important event of the year was the scientific expedition to Australia to study the conditions of apple transport to this country, particularly the ventilation and temperature of ships' holds, and the effect upon rate of ripening, formation of "brown heart," etc. The detailed results of the work are being published in separate reports, but it may be noted that four ships, representative of different systems of refrigeration, were fitted with distant-reading thermometers, electrical carbon dioxide indicators, and sampling tubes for gas analysis, and one of the most striking facts revealed was the extent to which accidental ventilation may occur in the ships' holds. Details are given of the work carried out during the year by the several Committees, represented by Sections I. to VII.

SECTION I. THEORY OF FREEZING.—The work on eggs has been continued, and it has been found that the yolk may be preserved without change, either by not allowing the temperature to fall below −6° C., as the irreversible change in consistence appears to be due to change in the nature of the coagulation and to occur between a temperature of −6° C. and some lower limit, or by hurrying over this critical interval sufficiently rapidly. In order to begin to elucidate these complicated changes a study was made of the way water is held by simpler colloid systems such as gelatine.

SECTION II. FISH PRESERVATION COMMITTEE.—The preservative action of ice containing small quantities of disinfectant was not very satisfactory in its results, owing to the disinfectant impregnating the flesh. The difference found between the autolysis of mutton and beef rapidly frozen in brine and in air, was found to be similar in the case of cod, and due to the fact that in the fluid set free in the case of the slowly frozen muscle (or the "drip") chemical changes occur more rapidly than in the remaining tissue.

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* Copies of the Report can be obtained from H.M. Stationery Office, Adastral House, Kingsway, W.C.2. Price 3s. net.

SECTION III. MEAT COMMITTEE.—Work has been continued on the succinic acid and carbohydrate metabolism of muscle. The presence of a factor in the pancreas (destroyed at 37° C.) that inhibits the formation of lactic acid in chopped muscle has been proved. The first series of studies on the growth of *Staphylococcus aureus* in meat extract has been completed, and problems connected with eggs, such as loss of weight on storage, and handling, have been investigated.

SECTION IV. ENGINEERING COMMITTEE.—The Australian Expedition necessitated the construction of a series of special instruments, and particular attention was devoted to the design of direct reading wet and dry hygrometers, micrometer hair hygrometers, absorption apparatus for the determination of carbon dioxide, apparatus for observing the ripening process in apples, temperature-measuring instruments, waterproof junction box, and a mercury in steel recording thermometer. Among the materials tested for thermal conductivity may be mentioned a hard form of expanded rubber with a surface resembling ebonite and a cellular interior. Work on the moisture content of materials in equilibrium with air of various humidities demonstrated the very slight capacity of slag wool for absorbing water vapour. When used as a heat insulator, however, this material may become coated with ice owing to the penetration of warm humid air into the insulation, which may occur in any granular or fibrous material.

SECTION V. FRUIT AND VEGETABLE COMMITTEE.—The work of this Committee is now very wide and varied, and it is only possible to indicate its scope. Great importance is attached to the commercial cold storage trials and general conclusions on three seasons' trials are now available. The knowledge gained on the Australian Expedition is being used in the continued investigation of the conditions of temperature and humidity in cold stores for this country. Work at the Low Temperature Research Station at Cambridge includes the study of temperature and metabolic balance in living plant tissues, respiration of apples, oxidases, and the physiology of fungal infection. At the Imperial College the following apple problems have been studied: acidity and nitrogen content in relation to storage life, concentration of sugars and the pectin content during storage and parasitism. The work on the chemistry of the cell wall has also been continued.

SECTION VI. OILS AND FATS COMMITTEE.—The examination of the series of glycerol methyl esters has been completed, so that the constitution of any mixed glyceride can now be determined, and a paper suggesting corrections in the published chemistry of the dichlorhydrins of glycerol is in preparation. Glycerol has been found to show little tendency to form either β- or γ-glucosides, and Fischer's glyceryl glucoside has been shown to be the butyleneoxide form. An investigation into the constitution of glycogen has been started. In the course of the continued work on the formation of fat by yeasts, directly extracted fat was found to contain the growth factor, and since the yeast was grown in the absence of direct sunlight it appears probable that the direct synthesis of the vitamin is being dealt with.

SECTION VII. CANNED FOOD COMMITTEE.—Work at Cambridge indicates that the determination of such final products of degradation as volatile organic bases, total acid radicles and amino acids gives some measure of the extent of ageing of fish under ordinary conditions of storage.

The Report is illustrated by numerous plates, charts and graphs. D. G. H.

New South Wales.

DEPARTMENT OF PUBLIC HEALTH.

ANNUAL REPORT OF THE GOVERNMENT ANALYST FOR THE YEAR 1923.

THE system for the control of the sale of food and drugs in New South Wales differs materially from that in force in Great Britain, practically the whole of the chemical work being carried out in the Government Laboratory. The Public Analysts in this State look to the general public for their practice, whilst analysts in the employ of the Government are not allowed to undertake private practice.

During the year 18,197 samples were analysed, of which number 16,183 were submitted under the regulations of the Pure Food Act, 1908, and 2014 in connection with the public services of the State.

MILK.—Of the food samples, 12,906 were milks from the metropolitan area, and 2496 were milks from country districts. In Sydney and suburbs 3·6 per cent. of the milk samples were adulterated, and of the country milks 9·1 per cent. did not comply with the standard. With the exception of 30 samples that contained formalin and 3 samples coloured with annatto, the adulterations were deficiencies in fat and in solids-not-fat. The percentage of adulteration in food and drugs other than milk was 29·4.

BRINE.—Fifty-two samples of brine, intended for use in salting meat, were examined, and 17 of these were condemned as unfit for the purpose. The figures obtained for the amounts of dissolved organic matter did not furnish a true indication of the degree of impurity, and the best practical test was found to be that of odour, which apparently varied with the age of the sample. It seems reasonable to demand that only brine not previously in use should be used for salting brine, or, if this is impracticable, that no brine should be allowed to remain in use for more than a day.

BUTTER.—A number of New Zealand butters failed to reach the standard for milk fat prescribed in this State. The deficiency was not great, and may be accounted for by the fact that the New Zealand standard requires only 80 per cent. of fat, as compared with 82 per cent. demanded by New South Wales.

Three samples of butter preservative composed of a mixture of borax and boric acid, contained 20, 50 and 120 parts per million of arsenic, respectively.

COFFEE.—One sample of coffee essence was over 50 per cent. deficient in caffeine. In connection with the general standard for coffee, a suggestion has been put forward to alter the regulation, which at present permits the use only of the seed of *Coffea Arabica* or *Coffea Liberica*, to include other genuine beans, particularly the *Robusta* bean. In order to ascertain whether this bean would conform with the requirements of the present standard as regards composition, samples were procured and analyses made. Six samples of the green bean were found to possess a fat content ranging from 9·5 to 10·8 per cent. (average 10·2 per cent.), and a caffeine content varying between 1·7 and 1·9 per cent. (average 1·78 per cent.). Eight samples of the roasted bean (5 whole and 3 ground) had a fat content of from 10·2 to 11·7 per cent. (average 10·8 per cent.), and a caffeine content ranging from 1·8 to 2·1 per cent. (average 1·9 per cent.). As a result of this examination it is apparent that, in comparison with published analyses of other varieties of the coffee bean, the *Robusta* bean has a high caffeine content, while its composition generally conforms to the N.S.W. standard for genuine coffee.

TEA.—Three samples of tea contained small quantities of arsenic, and a number of samples labelled "Golden Tips" contained only very small amounts, or, in some cases, no "golden tips."

GELATIN.—Three samples of powdered gelatin each contained 1/50 grain of arsenic per lb. The N.S.W. standard does not allow any metallic contamination of gelatin.

SAUSAGES.—Twenty-five samples were adulterated by the excessive use of the permitted preservative. Three samples of Devon sausage contained an excess of salt petre, and one sample of meat had been illegally preserved with formalin.

DRUGS.—Fourteen samples of acetyl salicylic acid tablets contained free salicylic acid, which is contrary to the B.P.

Artificial Carlsbad salts were found to consist of sodium sulphate instead of a mixture of salts.

A "non-poisonous" chlorodyne did not contain morphine (as prescribed by the Codex), but the presence of chloroform rendered the statement false and misleading.

CRIMINAL INVESTIGATION.—The Police submitted 135 exhibits in connection with criminal investigations. These included foods for examination as to the presence of poisonous or harmful ingredients, some of which gave negative results on analysis, whilst in other cases strychnine, arsenic, caustic soda, and ground glass, respectively, were found to be present. Ten samples of pills and medicines used for abortifacient purposes were examined, and exhibits were also submitted in connection with incendiary outbreaks, counterfeit coining, opium smoking, cocaine selling, illegal sale of spirituous liquors, laying of poison baits, sheep stealing (alteration of brands), quack medicine selling, etc.

Forty-four exhibits of human viscera were examined in connection with coroners' inquests. The poisons responsible for death consisted of cyanide, strychnine, arsenic, veronal, morphine, and opium. In one case the death of a child was due to the consumption of alophen pills, the viscera being found to contain phenolphthalein, aloin, and traces of strychnine and atropine. In another case, a woman's death was caused by the mistaken use of arsenic for baking powder, due to the fact that the arsenic was put in a tin labelled "baking powder" and with no other distinguishing mark.

THOMAS COOKSEY
(*Government Analyst*).

Toxicology and Forensic Chemistry.

Two lectures that contain much of interest to chemists in general were given last year, and have now been reprinted. The first, by Dr. H. Wilson Hake, F.I.C., was delivered at Westminster Hospital on September 30th under the title of "On Some Problems in Toxicology"; the other, by Sir William Willcox, M.D., F.I.C., was an address on "The Influence of Chemical Research on Medicine and Forensic Chemistry," to a joint meeting of the Manchester and District Section of the Institute of Chemistry and other Manchester Scientific Societies, on November 7th.*

Dr. Hake gives a comprehensive summary of the rise of toxicology, as a branch of science which only dates back to the time of Majendie (1783–1855) and of Orfila

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* Issued as a pamphlet by the Institute of Chemistry, Jan. 1925

(1787–1853), Professor of Medical Jurisprudence at Paris. The modern systematisation of the methods of examination is mainly due to Christison, Professor of Medical Jurisprudence (1822), whose "Treatise on Poisons" (1829) was the first work to direct attention to such points as the interval between the administration of a poison and the onset of the symptoms, the comparison of the symptoms with those of disease, the morbid appearances, and what he termed the *moral evidences*.

Christison was of opinion that modern chemical methods would make secret poisoning impossible, but Stevenson, fifty years later (1894) took a different view, pointing out that "many cases of chronic poisoning have been overlooked by medical practitioners, and death certificates signed from normal causes."

Three cases, to mention no others, within the last 20 years afford ample evidence that there is still a risk of mistaken diagnosis in cases of murder—*The Chapman Case* (1903). It was diagnosed that the victim was suffering from some form of intestinal trouble, whereas it was found, after *post mortem* examination, that 25 to 30 grains of tartar emetic were present. The *Seddon Case* (1912). There was a diagnosis of epidemic diarrhoea, but the symptoms were afterwards proved to have been produced by arsenic. *The Armstrong Case* (1922). Heart disease and kidney trouble were diagnosed, and the symptoms were subsequently proved to be due to arsenical poisoning.

It is suggested that insufficient use is made of chemical examination of the urine when the medical practitioner is in doubt as to his diagnosis, and when the possibility of poisoning cannot be excluded.

The lecture includes classified statistics of the poisoning cases for the 20 years ending with 1904, and for the period from 1914 to 1923.

Sir William Willcox, in his address, first deals with the development of chemistry, particularly in its relation to medicine, and gives a brief outline of the various chemical methods that have been devised as means of diagnosis by various diseases, such as micro-chemical processes for the examination of the blood, and gastric analysis after test meals.

In his account of the development of the methods of toxicological analysis he points out how much is owing to the work of Stevenson.

In various cases the application of newly-discovered tests for alkaloidal and other poisons has been of the greatest importance. In 1855 the expert for the Crown in the Palmer case was unable to say that strychnine was present, although there was little doubt that death was due to strychnine poisoning, and the prisoner was convicted. At the present time he would probably have been acquitted unless proof had been brought of the presence of the poison.

In the *Crippen Case* the poisoner had made use of hyoscine, which had previously been stated to be a decomposable alkaloid which no one would be able to detect after the lapse of a few days or weeks. But he had buried the viscera in quick-lime, thereby preserving them, and so probably enabling the hyoscine to be detected. Since that case the work of the lecturer has been checked by other chemists, who have found that it is possible to extract and identify hyoscine from viscera after a period of six months.

The electrolytic Marsh-Berzelius test for arsenic was first used by the lecturer in the Seddon case. For the first time, too, the organs of the body were separately weighed, and the arsenic determined in each, and from the results an estimate of the total arsenic in the body was formed.

In dealing with forensic evidence many valuable hints, based on long experience, are given. Stress is laid on the point that expert evidence should be free from all trace of bias. It is a great advantage for an expert witness to imagine himself in the position of the judge or cross-examining counsel, and his success as a witness will be much assisted by appreciating the psychological attitude of those who question him; it will also make him more tolerant of the searching questions put to him in cross-examination. Ambiguity and lengthy explanations should be avoided, as well as statements that are not answers to questions.

Referring to questions that may be put from books or reports, the lecturer suggests the wisdom of asking to see the work cited before giving a reply, and he concludes with the following advice, which sums up the whole question of expert evidence: "It is the duty and privilege of the expert witness to assist justice by his experience and advice. It is his duty to ensure that his views are clearly put before the Court and that not one jot or one tittle should be withheld or left in doubt."

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Influence of Glucose and Fructose on the Rate of Hydrolysis of Sucrose by Invertase from Honey. J. M. Nelson and C. T. Sottery. (*J. Biol. Chem.*, 1924, **62**, 139–147.)—Nelson and Cohn (*J. Biol. Chem.*, 1924, **61**, 193) found an increase in the rate of hydrolysis of sucrose by invertase from honey at the beginning of the reaction, which is unusual, since invertase action, when the invertase is obtained from bottom yeast, does not show this effect. Experiments are described which were carried out with the purpose of making a further study of this effect. Tables and curves show the results. Mutarotated glucose, besides retarding the action of a preparation of invertase from honey, when present in higher concentrations, also has an accelerating influence when present in lower concentrations. α-Glucose retards or accelerates, depending upon its concentration, less than the β or mutarotated forms. Mutarotated and β-fructose do not retard or accelerate to the same extent that the glucose does. There is little or no difference between the relative effects of the various forms of fructose. The presence of added glucose tends to eliminate the characteristic initial increase in the rate of hydrolysis of a 10 per cent. sucrose solution by invertase from honey. There appears to be a relationship between the relative concentrations of glucose and sucrose in respect of the extent of the influence of the hexose on the rate of the hydrolysis. P. H. P.

Nutritive Value of the Snail. (***Helix pomatia*, L.**) **L. Leger.** (*Comptes rend.*, 1924, **179**, 1622–1625.)—The part eaten (*a*) includes the well-developed foot, the head, stomach, rectum and oviduct, whilst that discarded (*b*) is chiefly the fat, liver and albuminous glands. The composition at the end of hibernation is as follows, calculated to percentages on the dry basis:—Proteins, (*a*) 79·98, (*b*) 63·25; ether extract, (*a*) 2·12, (*b*) 6·50; mineral matter, (*a*) 8·27, (*b*) 8·37; extractives, (*a*) 9·63, (*b*) 21·88, including (*b*) reducing and invert sugars 2·74 and glycogen (Frankel-Garnier method) 3·07 per cent. All the glycogen and reducing sugars, the greater part of the nutritive substances, and the most digestible material is found in the discarded portions. D. G. H.

Rancidity and the Kreis Test. T. W. Jones. (*J. Soc. Chem. Ind.*, **1924, 43,** 1258–1259.)—A review of the history of the Kreis test and its various modifications suggests that Powick's modification of the Kreis test of Kerr for the detection of acrolein may be the long-sought specific test for rancidity. In this test 1 drop of 3 per cent. hydrogen peroxide solution is added to 1 or 2 drops of the diluted acrolein solution, followed, after 1 minute, by 5 c.c. of hydrochloric acid (sp. gr. 1·19), and after shaking, by 5 c.c. of an ethereal solution of phloroglucinol, and the mixture again shaken. The hydrochloric acid assumes a deep red colour, and a spectroscopic examination shows a fairly narrow absorption band in the yellow green region. This test is positive with rancid fats, but not with vanillin, eugenol, cinnamic aldehyde and non-rancid cotton seed oil, all of which react with the Kreis reagent, and it is suggested that the substance responsible for the colour may be a product of the decomposition or, rather, the oxidation, of oleic acid, which is likely to prove identical with the acrolein and hydrogen peroxide complex. D. G. H.

The Glycerides of Cacao Butter. K. Amberger and J. Bauch. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48,** 371–390.)—An exhaustive investigation into the composition of cacao butter by methods depending on fractional crystallisation from acetone, ether and alcohol (*cf.* ANALYST, 1924, **49,** 283) has shown it to consist of the glycerides of oleic, stearic, and palmitic acids, the acids being present in the following proportions:—Oleic acid, 43–45 per cent.; palmitic acid, 23–25 per cent.; and stearic acid, 31–33 per cent.; no acids of higher molecular weight could be detected. The following proportions of glycerides were found:—Tristearin, 0·02; β-palmito-α-α-distearin, 0·03; oleo-α-β-distearin, 24·92; oleo-β-palmito-stearin, 20·29; and α-palmito-α-β-diolein, 54·74 per cent. A sample of the fat which had been hydrogenated in the presence of palladium had an iodine value of 5·9 and m.pt. 60·5° C., and contained 77 per cent. of palmitic acid with 22·7 per cent. of stearic acid, and the separated glycerides consisted of tristearin, 25; β-palmito-α-α-distearin, 20; and α-palmito-α-β-distearin, 55 per cent. H. E. C.

Characterisation and Evaluation of Ferric Casse in Wines. P. Malzevin and J. C. Essner. (*Ann. Falsificat.*, 1924, **17,** 473–477.)—The following procedure serves for the determination of the proportion of ferrous iron susceptible to oxidation present in wine. A certain volume of the wine is treated with a few c.c. of hydrogen peroxide and with excess of 5 per cent. tannin solution, which is added until no further blackening occurs. This wine is then added gradually from a burette to 5 c.c. of 1 per cent. citric acid solution contained in a porcelain dish; at first a black spot forms and then disappears, and the addition is continued until the black spot does not disappear, but renders the liquid of similar colour to the added wine. If the volume of wine run in from the burette is n c.c., the quantity of "cassable" iron per hectolitre or the degree of "cassability" of the wine is equal to $38/n$ grms., and the amount of crystallised citric acid to be added to a hectolitre of the wine to preserve it from ferric casse is $\frac{12{\cdot}76 \times 38}{n}$.

In France, citric acid may be added only to the extent of 50 grms. per hectolitre, which corresponds with only 4 grms. of "cassable" iron, an amount often surpassed. In such case the wine may be treated with an amount of tannin equal to four times that of the "cassable" iron present, and then oxygenated, fined, and filtered; the degree of "cassability" is then determined again, and the wine treated with the suitable quantities of tannin and citric acid, as described above. Ferric casse begins to be troublesome when the degree of cassability equals 2, is very active when the value is 4, and is difficult to combat if the value reaches 8 to 9. Red wines may be treated in the above manner, but the titration must be carried out in a good light in order that the end-point may be ascertained. T. H. P.

Determination of Adrenaline in Suprarenal Powders by an application of the Denigès-Grimbert-Leclère Reaction. O. Bailly. (*J. Pharm. Chim.*, 1924, **30**, 404–405.)—One grm. of suprarenal powder and 1 c.c. of N sulphuric acid are shaken up with distilled water, and after 15 minutes made up to 100 c.c., and allowed to remain in contact another 15 minutes before filtering. Two c.c. of the filtrate are added to a solution of 1 grm. of sodium acetate in 8 c.c. of water, shaken, and 3 drops of a 5 per cent. solution of mercuric chloride added. The resulting red colour attains its maximum intensity in 3 minutes, and can be compared with that of a solution containing 0·01 grm. of adrenaline and 1 c.c. of N sulphuric acid in 100 c.c. of water. D. G. H.

Colorimetric Determination of Solutions of Novocaine. P. Chertmy. (*J. Pharm. Chim.*, 1924, **30**, 408–411.)—A type solution and the solution to be examined (say 1 grm. in 200 c.c. of water in each case) are diluted 100 per cent., and to 5 c.c. of each is added, if necessary, 1 drop of 0·1 N iodine solution in order to eliminate any colour due to adrenaline; the solutions are then diazotised by adding 1 c.c. of dilute hydrochloric acid and 3 drops of 10 per cent. sodium nitrite solution. On adding 10 c.c. of 2 per cent. β-naphthol in N sodium hydroxide solution a red colour develops, which is matched in the two solutions by suitable dilution. Novocaine solutions are not appreciably altered if sterilised in slightly alkaline glass ampoules at 100° C., but after sterilisation at 120° C. a good glass should be used; or a 2 per thousand solution of benzoic acid may be added to the novocaine. D. G. H.

Biochemical, Bacteriological, etc.

Fat-Soluble Vitamins. XX. Modified Technique for the Determination of Vitamin A. H. Steenbock, M. T. Nelson and A. Black. (*J. Biol. Chem.*, 1924, **62**, 275–287.)—Absence of growth on suitably constituted rations as used at present cannot be taken as an indication of the absence of vitamin *A* unless the antirachitic factor is supplied. A method has therefore been devised in which light is used as the antirachitic agent for vitamin *A* determination. A Cooper-Hewitt BY type quartz mercury vapour lamp was used as a source of light for irradiation. To this, rats were exposed at a distance of about 22 inches for 10 minutes daily, 6 days of the week. Charts and tables show the results of the experiments. When vitamin *A* is present in sufficient amounts the amount of antirachitic vitamin can also be determined in foods, provided the inorganic relations are not seriously disturbed. It has been concluded that hog millet, and probably common millet as well, contain considerable quantities of vitamin *A*. Both, however, are deficient in the antirachitic factor, which is responsible for their failure to support growth when supplemented with casein and the ordinary salt mixture. Alfalfa carefully cured in the dark was found very rich in vitamin *A*. Even 0·5 per cent. supported normal growth when the antirachitic factor was supplied in the form of light, otherwise 4·0 per cent. was required. When cured in the sun, exposed to dew and rain, vitamin *A* was destroyed, but an antirachitic activation of certain substances in the alfalfa is suggested. P. H. P.

Critical Study of the Jendrassik Reaction for Water-Soluble Vitamin *B*. V. E. Levine. (*J. Biol. Chem.*, 1924, **62,** 157–161.)—The ferric ferricyanide reaction proposed by Jendrassik (*J. Biol. Chem.*, 1923, **57,** 129) is not a specific test for water-soluble vitamin *B*. The method consists in adding acetic acid to the concentrated aqueous solution of the preparation to be tested for vitamin *B* to make a 2 per cent. solution, then adding freshly prepared ferric ferricyanide. In the presence of water-soluble vitamin *B*, a characteristic blue colour is developed, the result of the formation of ferric ferrocyanide or Prussian blue. The ferricyanide in the reagent is reduced to ferrocyanide which further reacts with the ferric chloride. The reagent is added as long as the depth of the colour increases. The tube containing the reaction mixture is stoppered to hinder reoxidation, allowed to stand for 10 minutes, and its contents diluted with 1 to 5 volumes of distilled water, and again observed for colour. A positive test is indicated by the presence of a distinct blue colour, or, after standing for some time, of a blue precipitate; a negative one, according to Jendrassik, by lack of change or the formation of a green colour. The reaction is given by phenols, and may serve as a test for them in the absence of a few positively reacting non-phenolic compounds. The list of substances examined is given. Alkali reacts with phenols to give derivatives or fractions that no longer respond to the ordinary phenol tests. Phenols resemble vitamin *B* with respect to the destructive effect of alkali. The fact that active preparations of water-soluble vitamin *B* do not give a positive Millon or Liebermann reaction is no proof that vitamin *B* is not a phenol, since neither of these two reactions is given by all phenols. The work does not preclude the probability of the presence in the vitamin *B* molecule of one or more phenolic groups. P. H. P.

Vitamin Potency of Cod Liver Oils. A. D. Holmes. (*Ind. Eng. Chem.*, 1924, 16, 964–965.)—Comparative feeding tests with freshly rendered cod liver oil and that obtained from livers which had rotted for eight months showed that the latter oil possessed less than one-twelfth of the vitamin potency of the fresh oil. The rotting process also altered the physical and chemical characters of the oil; the specific gravity increased from 0·9210 (fresh oil) to 0·9548, the refractive index from 1·4775 to 1·4795, the saponification value from 187·3 to 206·8, and the free fatty acids from 1·3 to 23·3 per cent. The iodine value decreased from 151·4 to 112·9. W. P. S.

Detection of Pollution in Shell-fish. J. W. H. Eyre. (*Public Health*, Dec., 1924; *Lancet*, 1924, II., 973.)—Since disease-producing "intestinal" bacteria rarely occur in shell-fish except in association with a preponderance of *B coli*, a bacteriological examination of a batch of oysters will enable an estimate to be made of their safety. In view of the vexatious delay, from the point of view of the oyster merchant, of the ordinary method of estimating the number of *B. coli*, a "short method" has been devised, by means of which it is possible to form an opinion within 24 hours. The oysters are opened with aseptic precautions, and tubes of bile-salt broth, glucose broth and litmus milk, in suitable dilutions, are inoculated with the liquor and incubated for 24 hours, after which the presence or absence of *B. coli* is ascertained by observation of the usual changes in the culture media. If, for example, acidity and gas are present in the bile-salt broth culture, the inference is drawn that the oyster from which the liquor was derived contained *at least* 50 *B. coli*, and is therefore to be condemned after being tested by the more tedious method of examination.

When a batch of shell-fish is condemned the Company of Fishmongers, which has control over the London fish market, usually takes action, since it is a much more involved process for any action to be taken by the local health authorities.

Anaerobic Bacteria in Meat Foods and their connection with Gastro-intestinal Disturbances. — Brekenfeld. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, 48, 174–175.)—Although anaerobic bacteria do not produce the paralytic manifestations characteristic of true botulism, it is thought that they are often responsible for the milder types of food poisoning producing gastro-intestinal troubles. This is borne out by investigation of some cases of illness caused by sausages; the bacteria isolated included cocci, *B. subtilis*, and *B. coli*, and certain anaerobes, Frankel's gas bacillus, pararauschbrand (paratyphoid?), and the wart-bacillus (Warzen bacillus). The *B. coli* and other types are common in sausages, and are not likely to occasion sickness, *B. subtilis* is not pathogenic, and it is probable that the gastric symptoms were due to the anaerobes. It is desirable that special attention should be given to the examination of the anaerobic bacteria in the investigation of meat foods which have caused illness. H. E. C.

Determination of Hydrogen Sulphide in Bacterial Cultures and in certain Canned Foods. C. R. Fellers, O. E. Shostrom and E. D. Clark. (*J. Bact.*, 1924, **9**, 235–249; *Chem. Abstr.*, 1924, **18**, 2731.)—The hydrogen sulphide is drawn from the acidified liquid through standard iodine solution by means of a current of air, and any iodine volatilised is caught in a standard solution of sodium thiosulphate. The hydrogen sulphide is then determined by titration of the combined iodine and thiosulphate solutions. From 99 to 100 per cent. of the hydrogen sulphide present is recovered by 15 minutes' aeration. Ordinary bacterial products such as volatile fatty acids, phenols, skatol, indol, etc., and small amounts of ammonia do not affect the results. Of 53 organisms tested, 12 gave positive results with Difco peptone. Armour's and Witte's peptone were not satisfactory for the test.

Bacterial Flora of Salt. Rappin and Grosseron. (*Ann. Chim. anal.*, 1924, **6**, 353–355.)—Refined salt from various sources was found to contain up to 8300 bacteria and 400 mould (spores) per grm. and samples of crude salt from 6000 to 76,000, and even 300,000 bacteria and 100–700 mould (spores), and although the majority of the organisms were saprophytic, toxic cultures were obtained in some cases. Brines of various ages and sources were also found to be badly contaminated, and there is little doubt that the salt may constitute a serious source of danger when added to food products. For food preservation purposes the salt should be free from bacterial contamination and be prepared under strictly aseptic conditions. D. G. H.

Microchemical Test for Nucleic Acid of the Thymonucleic Acid Type. R. Feulgen and H. Rossenbeck. (*Z. physiol. Chem.*, 1924, **135**, 203–248; *Chem. Abstr.*, 1924, **18**, 2725–2726.)—The "nucleal" reaction consists in partial hydrolysis of the preparation on the microscope slide by treatment with *N* hydrochloric acid at 60° C. for 4 minutes, and then for 1 to 3 hours at the ordinary temperature, after which the material is washed with water and immersed in Schiff's reagent (magenta and sulphurous acid). An intense violet coloration, due to the formation of an aldehyde by the hydrolysis, is produced in the case of nucleic acid from animal sources or from higher plants, whereas the pentose-containing nucleic acids of yeast and bacteria do not show the reaction. Any possibility of confusion due to the presence of naturally occurring aldehydes is eliminated by the fact that nucleic acids do not give the reaction before hydrolysis. Moreover, the aldehydic grouping responsible for the reaction cannot be extracted with water. No reaction is obtained with the lowest forms of organisms, including yeasts and trypanosomes, but reactions are produced by infusoria, wheat germs, pus, spermatozoa, and nucleated cells of animals in general. The cell structure remains intact during the test, and the stain can be readily observed in the nucleus.

Detection of Poisonous Gases in Blood. E. Kohn-Abrest. (*Comptes rend.*, 1924, **179**, 903–906.)—A method is described for the detection of alcohol, hydrogen sulphide, hydrocyanic acid, carbon dioxide, and carbon monoxide in blood, use being made of a modified form of the apparatus previously described. (See Ogier and Kohn-Abrest, *Traité de Chimie toxicologique*, 2nd Edition, Vol. I., 1924.) T. H. P.

Normal Occurrence of Carbon Monoxide in Blood. M. Nicloux. (*Comptes rend.*, 1924, **179**, 1633–1636.)—Contrary to the statement of Buckmaster and Gardner (*Proc. Roy. Soc.* 1909, **81** [B], 515) carbon monoxide is a normal constituent of blood and may be withdrawn from it either by subjecting it to reduced pressure; by means of phosphoric acid, by displacement with nitrogen peroxide, or in the living subject by pure oxygen. It may be recognised as carbon monoxide: By forming with hæmoglobin (1) a compound resistant to decomposition *in vacuo*, and (2) one which shows the two characteristic absorption bands of hæmoglobin-carbon monoxide. It also gives similar results when analysed by means of the micro-eudiometer. The amount of carbon monoxide found in the blood of the ox, pig and horse was about 0·1 c.c. per 100 c.c. of blood. D. G. H.

Method for the Determination of Lipoid Phosphorus in Blood and Plasma. J. C. Whitehorn. (*J. Biol. Chem.*, 1924, **62**, 133-138.)—The method of Bell and Doisy (*J. Biol. Chem.*, 1920, **44**, 55) for determining inorganic phosphates and the procedure of Randles and Knudson (*J. Biol. Chem.*, 1922, **53**, 53) were tried, but could not be satisfactorily utilised for determining the lipoid phosphorus of blood plasma. A method has been devised for this which is not so simple, but which is believed to be more reliable. The chief difficulties encountered were in the control of the digestion process, and these have been overcome without giving up the use of nitric acid. Errors due to local overheating have been avoided by the use of enough sulphuric acid to fill the hemispherical end of the test-tube (1 c.c. of specific gravity 1·84), and the traces of nitric acid remaining after digestion are eliminated by sulphur dioxide liberated from sodium sulphite. The use of so much acid also reduces to relative insignificance the amount of sulphuric acid volatilised during digestion. The intense acidity of the digestion product has necessitated a departure from the process of colour development described by Bell and Doisy or modified by Briggs (*J. Biol. Chem.*, 1922, **53**, 13). With this degree of acidity, 10 minutes in boiling water after the addition of acid molybdate, sodium sulphite and hydroquinone, produces a clear blue colour, the intensity of which, after cooling, is proportional to the quantity of orthophosphoric acid, even when the difference exceeds 33 per cent. A blank determination gives only a faint yellow colour. The blue colour increases slowly for about 24 hours and does not disappear for several weeks. Several unknown solutions can be compared with the same portion of standard in the colorimeter. If the blue solution is diluted with distilled water the intensity of colour varies in inverse proportion to the dilution. Unexpectedly deep colours may therefore be adjusted nearly to the colour of the standard. Examples given show the method to be extremely accurate and reliable. P. H. P.

The Passage of Boric Acid through the Skin by Osmosis. L. Kahlenberg. (*J. Biol. Chem.*, 1924, **62**, 149–156.)—The author has shown, after studying many substances such as lithium chloride, caesium chloride, rubidium chloride, strontium chloride, borax and lithium borate, that boric acid, and this alone, passes through the living skin in perceptible quantities. When salts of boric acid, like borax and lithium borate, are used, no boric radical is found in the urine. The amounts of boric acid found in the urine of a normal person whose feet are soaked in boric acid solution are much the same (a few hundredths of a per cent.) as those observed in patients treated with boric acid compresses for blood-poisoning. In 5 minutes after the feet are immersed in the boric acid solution, boric acid is present in the urine. All the substances tested pass readily through dead human skin and also through other dead animal membranes. Since dead skin and living skin act differently osmotically, one is led to the conclusion that they are chemically different. Similarly, living mucous membranes are chemically different from the living skin. Boric acid given by the mouth to patients suffering from blood poisoning does not quell the infection, but relatively large quantities of it are found in the urine. Sulphuric, hydrochloric and citric acids, when taken by mouth, acidify the urine, whereas when the skin is bathed with them the first two cause an alkaline reaction of urine, whilst citric acid has the opposite effect. Therefore it does not seem so peculiar that boric acid should have quite a different effect when taken internally from that produced when it passes into the system through the living skin. P. H. P.

Toxicological, Forensic, etc.

Effect of Lead on Blood Cells. J. C. Aub, P. Reznikoff and D. E. Smith. (*J. Exptl. Med.*, 1924, **40**, 151–208; *Chem. Abstr.*, 1924, **18**, 2922–2923.)—Previous work has indicated that the anæmia observed in cases of lead poisoning is due to destruction of blood rather than to diminished production of blood. In studying this phenomenon the blood cells were poisoned with lead *in vitro*, and distinct effects were obtained, even when only 0·001 mgrm. of lead was added to approximately 5 billion washed red corpuscles. By the addition of 0·01 mgrm. such a marked resistance to hypotonic salt solution develops that complete hæmolysis does not occur until the cells are exposed to a saline solution of 0·05 per cent., whereas untreated cells are completely hæmolysed in 0·25 to 0·225 per cent. solutions. The reaction is quantitative, and varies with the concentration of lead used. The effects with arsenic are very slight in comparison. The change in hæmolysis does not appear in the blood of all species of animals. In the species in which it does occur, anæmia and stippling of the red cells also tend to develop readily during lead poisoning. *In vitro* cells that have been exposed to lead are more fragile than normal blood cells. It has been found that inorganic phosphate in the same concentration as is normally present in the serum neutralises the same quantity of lead as does the whole serum itself. The addition of inorganic phosphate to red blood cells or to whole blood greatly reduces the action of lead upon the cells, but cannot eliminate it completely, indicating that there is a simultaneous reaction between the lead and serum and lead and corpuscles when all are present. In agglutination experiments with colloidal iron and arsenic a marked difference was observed between "leaded" and normal cells.

Lead Tetraethyl Poisoning. H. Leffmann. (*Amer. J. Pharm.*, **1924, 96,** **861–863.**)—Attention is directed to the occurrence of numerous very severe cases of poisoning caused by exposure to the fumes of lead tetraethyl, which has recently been introduced as a remedy for "knock" with petrol (gasoline).* This has led to drastic regulations being made by various U.S.A. public health authorities, and the petrol companies have accordingly withdrawn the product from sale pending further investigation to determine the limits of safety for its use. Tetraethyl lead, like the other organo-metallic compounds, produces spasm of the glottis when inhaled in admixture in large proportions with air, but when present in minute quantities, as in motor engine exhaust gases, it produces chronic symptoms of lead poisoning.

* According to Jolibois, when the lead tetraethyl is decomposed the rough places in the cylinder, which act catalytically in promoting the spontaneous explosion of the gaseous mixture, become coated with metallic lead, whereby the tendency to explode is reduced.—Editor.

Mercury Poisoning from Electric Furnaces. L. Jordan and W. P. Barrows. (*Ind. Eng. Chem.*, 1924, **16,** 898–901.)—Several cases of mercury poisoning occurring amongst men operating high-frequency induction furnaces led to the discovery that mercury vapours escaped from the mercury discharge gaps of the high-frequency converters. The air near the furnaces contained 0·7 mgrm. of mercury per cubic metre, a quantity sufficient to cause mercury poisoning if daily exposure is continued for several months. When a new type of discharge gap having stationary electrodes, mercury-sealed discharge chambers, and operating in an atmosphere of hydrogen, was installed and enclosed in a separate compartment fitted with a forced-draught hood, the escape of mercury vapours from the discharge-gap could not be detected. W. P. S.

Agricultural Analysis.

Copper Sulphate in Agriculture. E. Cerasoli. (*Giorn. Chim. Ind. Appl.*, **1924, 6, 536–537.**)—The proportion of copper sulphate in copper-lime mixtures used as fungicides may be diminished from 8 to 1 per cent. or even less, the efficacy of the treatment depending, not on a high content of copper in the material, but on the thoroughness of the application. The use of such substances in the form of powder is recommended, especially where water is scarce. T. H. P.

Nickel and Cobalt in Arable Land. G. Bertrand and M. Mokragnatz. (*Comptes rend.*, **1924, 179, 1566–1569.**)—Nickel and cobalt were found in all the samples of soil examined which were taken from various parts of France, Germany, Denmark, Italy, Rumania, and Serbia. The proportion of nickel varied between

5·5 and 38·6 mgrms. per kgrm. of dry earth, and that of cobalt between 0·3 and 11·7 mgrms., but the lower limits were exceptional in both cases. Probably the proportion of these metals present varies with the depth from the surface.

D. G. H.

Determination of Manganese in Agricultural Soils. G. Bertrand. (*Bull. Soc. Chim.*, 1924, **35**, 1522–1527.)—The air-dried soil is passed through a 1 mm. sieve, and the proportion of pebbles and fine soil ascertained. A sample of the latter is finely ground in agate, for the following tests. (*a*) *Manganese soluble in dilute acetic acid.*—0·5 grm. is boiled for a short time with 5 to 10 c.c. of one per cent. acetic acid in a boiling-tube. After settling, the clear solution is decanted through a filter; the residue is re-treated 3 to 4 times in the same manner, and the combined filtrates evaporated to dryness in a porcelain dish. The residue is calcined, dissolved in strong hydrochloric acid, and the solution evaporated to fumes with sulphuric acid. The cold mass is dissolved in dilute nitric acid, and the manganese determined colorimetrically. (*b*) *Manganese as humate.* The insoluble residue from (*a*) is extracted several times with ammonia (1 in 10 or 20 water) until the extract is only coloured straw-yellow; the united filtrates are treated as in (*a*). (*c*) *Manganese soluble in strong hydrochloric acid.* The residue from (*b*) [or *a*, if (*b*) is not required] is collected on the filter and the organic matter destroyed by ignition. The residue is digested at gentle heat with hydrochloric acid, and the liquid then evaporated to complete dryness. The residue is moistened with hydrochloric acid, water is added, and the solution is decanted through a filter; the residue is treated once or twice more in the same manner. The combined filtrates are evaporated to fumes with sulphuric acid, etc., as under (*a*). (*d*) *Total manganese.* The finely-ground sample is fused with sodium carbonate in a platinum crucible, the fused mass dissolved in hydrochloric acid, and the silica rendered insoluble by evaporation. The filtrate is evaporated to fumes with sulphuric acid for colorimetric determination. The manganese present in a refractory form is found by difference.

W. R. S.

Poisonous Alkaloid from the Transvaal *Homeria pallida*. M. Rindl. (*Trans. Roy. Soc., S. Africa*, 1923, **11**, 251–256; *Chem. Abstr.*, 1924, **18**, 2909.)—The yellow plant (*Homeria pallida*), both fresh and dried, is poisonous to stock, less than ½ lb. of the dried material being fatal to cattle. Alkaloids are present in all parts of the plant, but cyanogenetic glucosides and saponins are absent from the parts above ground. Alkaloidal reactions are given by the alcoholic extract only. The extraction and all other operations should be conducted in the cold, as far as possible, as these alkaloids are very sensitive to heat. On extracting the slightly ammonical aqueous solution with chloroform, a light yellow transparent varnish, with some crystals, is obtained. This active alkaloidal principle has a slightly bitter taste, is precipitated by the usual reagents, is insoluble in benzene, sparingly soluble in dry ether, and readily soluble in alcohol, acetone, ethyl acetate, chloroform and water. It has a pronounced reducing action. Its effect upon the heart resembles that of digitalis, and in large doses it is a cardiac poison. It is the only known alkaloid (with the exception of that in the bark of *Erythrophloem guineense*), which resembles digitalis in its effects, but is not a glucoside. No crystalline form of the alkaloid and no well defined derivatives have yet been obtained.

Organic Analysis.

Colour Reaction of the Alcoholic Hydroxyl Group. W. Parri. (*Giorn. Farm. Chim.*, 1924, **73**, 109–112; *Chem. Abstr.*, 1924, **18**, 2667.)—A reagent prepared by dissolving 3 grms. of phosphomolybdic acid and 0·3 grm. of ammonium vanadate in 100 c.c. of concentrated sulphuric acid gives colorations when heated with various alcohols, hydroxy acids, polyatomic acids and sugars, and allowed to stand. The most characteristic reaction is the azure blue colour obtained with monatomic alcohols, such as ethyl alcohol, secondary butyl alcohol, and *iso*-amyl alcohol (not methyl alcohol).

Differentiation of Citric from Tartaric Acid. W. Parri. (*Giorn. Chim. Ind. Appl.*, 1924, **6**, 537–538.)—The presence of a small proportion of citric acid in tartaric acid may be detected in the absence of certain other compounds (see preceding abstract), by a reagent prepared by warming 3 grms. of precipitated phosphomolybdic acid (or ammonium phosphomolybdate) and 0·3 grm. of ammonium vanadate in 100 c.c. of concentrated sulphuric acid; a deep blue coloration is formed, this becoming green on heating, but resuming its original colour on cooling. The reaction is disturbed if much sugar is present, owing to browning; also, dextrose or sucrose gives a green coloration. T. H. P.

Determination of Alumina in Chrome Leather. D. Woodroffe. (*J. Soc. Leather Trades Chem.*, 1924, **8**, 581.)—Two methods are given. (1) *With platinum crucible*:—The ash from 5 grms. of leather is fused in a platinum crucible with fusion mixture, extracted with excess of dilute hydrochloric acid, ammonium chloride and slight excess of ammonia solution are added, and the solution boiled till the precipitate flocculates. The aluminium hydroxide thus formed is then filtered off, washed with weak ammonia solution, dried, ignited and weighed.

(2) *Without platinum crucible*: The ash from 5 grms. of leather is fused gently in an iron crucible with 8 to 10 times its weight of pure sodium peroxide (A.R.) and kept fused for 3 minutes. The mass is dissolved in warm distilled water and filtered from the oxides of iron. The filtrate is acidified with hydrochloric acid, boiled with excess of ammonia, and the analysis completed as above. Experiments show the methods to work well on pure materials, but when tested on leather ash by different analysts the results were not quite in agreement, and further work is in progress. It is pointed out that the presence of alumina in chrome leather may be due to the use of alum in the tannage or to the presence of pigments in or on the leather. R. F. I.

New Simplification in the Official Method of Tannin Analysis. A. Jamet. (*J. Soc. Leather Trades Chem.*, 1924, **8**, 613.)—The long time required by the Official Method is a great objection to its use for works control. The various stages of determining non-tans have been specially studied, with the following results:—The time for chroming the hide powder has been reduced from one hour to fifteen minutes. The washing of the chromed hide powder can be reduced from one hour to fifteen minutes, if certain precautions are taken. Mechanical agitation at an increased speed (60–80 revolutions per minute) for chroming or detanning is not indispensable; shaking from time to time gives identical results. The hide powder after detannisation constitutes a perfect filter, provided that it rests on a very fine gauze (brass). A supplementary filtration with washed kaolin through paper therefore becomes superfluous. A special simple apparatus is described by means of which a clear solution of the non-tans can be obtained without a single transference of the hide powder. For evaporation 30 c.c. are taken instead of 50 c.c., thus reducing the time required. Comparative results obtained by this method and by the Official Method on chestnut extract, quebracho extract and oak bark are in close agreement. The total time required is reduced from 8 or 9 to 3 hours. R. F. I.

Fig Tree (Gondang) Wax. A. J. Ultee. (*Pharm. Weekblad*, 1924, 1118–1120.)—Fig tree wax, obtained from the sap of *Ficus variegata*, was shown by Greshoff and Sack (*Rec. Trav. Chim.*, 1901, **20**, 65) to consist of ficoceryl alcohol and ficoceric acid, the alcohol also being present in the free condition. The author has identified the ficoceryl alcohol as β-amyrol, it being present in the combined form as acetate, and the ficoceric acid as impure palmitic acid. Fig tree wax is therefore β-amyrol-palmitate, and can be prepared synthetically by boiling 1 mol. of β-amyrol dissolved in benzene, with 2 mols. of palmitic acid chloride for one hour, and subsequently heating the product in a porcelain dish for two hours on a boiling water bath. The dry residue is extracted with hot alcohol, and the wax crystallises out on cooling (m.pt. 77° C.). W. S. S.

Modified Method of Determining Cellulose in Wood. G. J. Ritter. (*Ind. Eng. Chem.*, 1924, **16**, 947–948.)—The chlorination periods mentioned in a previous paper (ANALYST, 1924, **49**, 196) may be reduced to three minutes without any appreciable difference in the yield of cellulose. Discoloration of the cellulose during drying has been ascribed to the use of permanganate as the bleaching agent and its subsequent incomplete removal, but discoloration also occurs when hydrogen peroxide or sulphur dioxide is used. It is recommended that the cellulose should be digested for two hours with hot water before the final washing; in this way the undesirable gelatinous property of cellulose is eliminated, filtration and washing are facilitated, and the cellulose contains a smaller quantity of substances soluble in hot water. W. P. S.

Determination of Sulphur in Mixtures of Raw or Vulcanised Rubber. V. C. Butironi. (*Giorn. Chim. Ind. Appl.*, 1924, **6**, 535–536.)—The following rapid method yields accurate results. About 20 grms. of pure potassium hydroxide are melted in a roomy silver crucible the bottom of which projects not more than 0·5 cm. through a hole in an asbestos card, and are then allowed to cool until pasty. The finely ground or rolled sample of rubber (0·2 to 0·5 grm.) is then introduced and immediately mixed in with a silver spatula so that it becomes completely coated with semi-fused potash; by this means any free sulphur on the surface of the sample is fixed. Gentle heating is continued until the destruction of the organic matter is almost complete, after which about 5 grms. of potassium nitrate are added. The subsequent procedure is that usually employed, any iron present in the resulting solution being removed as hydroxide before precipitation of the barium sulphate. T. H. P.

Titration of Aniline and its Homologues. D. O. Jones and H. R. Lee. (*Ind. Eng. Chem.*, 1924, **16**, 948–949.)—The amine is treated, in hydrochloric acid solution, with an excess of nitrite, nitric acid is added to prevent decomposition of the nitrite, and the excess of the nitrite is titrated subsequently. A quantity of the sample (the weight taken should be such that one-tenth of it will require about 40 c.c. of 0·1 *N* nitrite solution) is dissolved in 5 per cent. hydrochloric acid and diluted to 500 c.c.; 50 c.c. of this solution are diluted to 300 c.c., 25 c.c. of concentrated hydrochloric acid are added, the mixture is cooled to 0° C., and 50 c.c. of 0·1 *N* nitrite solution and 10 c.c. of concentrated nitric acid (free from nitrous acid) are added. After thirty minutes the excess of nitrite is titrated with standardised *p*-nitroaniline solution. W. P. S.

Analysis of Dehydrothio-p-toluidine Sulphonic Acid. H. R. Lee and D. O. Jones. (*Ind. Eng. Chem.*, 1924, **16**, 930–931.)—In the method proposed an alkaline solution of the amine is treated with an excess of nitrite, the mixture is then acidified, and the excess of nitrite is titrated with standardised primary amine solution. A quantity of 1·3 grms. of dehydro-thio-*p*-toluidine ammonium sulphonate is dissolved in 200 c.c. of water and 5 c.c. of 10 per cent. sodium hydroxide solution, the mixture is cooled to about 0° C., and pieces of ice are added. Fifty c.c. of 0·1 *N* sodium nitrite solution are then introduced, the mixture is stirred while 25 c.c. of concentrated hydrochloric acid and 10 c.c. of concentrated nitric acid (free from nitrous acid) are added, and, after ten minutes, the excess of nitrous acid is titrated with 0·1 *N* *p*-nitroaniline solution. The end-point is reached when a small drop of the mixture ceases to give a blue coloration immediately with a drop of starch iodide solution. Each c.c. of 0·1 *N* nitrite solution is equivalent to 0·033733 grm. of dehydrothio-*p*-toluidine ammonium sulphonate. The *p*-nitroaniline solution is prepared by dissolving 14 grms. of the substance in 200 c.c. of hot water and 150 c.c. of concentrated hydrochloric acid, filtering the solution after fifteen hours, and diluting the filtrate to 1 litre; it is standardised against pure sulphanilic acid. W. P. S.

The Optical Properties of Some Amino Acids. G. L. Keenan. (*J. Biol. Chem.*, 1924, **62**, 163–172.)—A study of the optical properties of many of the known crystalline amino acids was made by the immersion method commonly used by crystallographers and mineralogists and more recently successfully applied by Wright (*J. Am. Chem. Soc.*, 1916, 38, 1647) and Wherry (*U.S. Dept. Agric., Bull.* 679, 1918) to the study of synthetic inorganic and organic crystalline substances. The oily liquids, in which the acids are insoluble, best suited for determining the optical properties by this method were found to be mixtures of mineral oil (Squibb's) with $n=1{\cdot}49$, monochloronaphthalene with $n=1{\cdot}64$, monobromonaphthalene with $n=1{\cdot}66$, and, in a very few cases, methylene iodide with $n=1{\cdot}74$. These oils were mixed in such proportions that each differed in n from the next by 0·005 and their exact n values were determined on a refractometer. Observations were made in yellow light, approximating that of the D line. In addition to the optical determinations, photomicrographs were made of the amino acids to illustrate their crystal habit as ordinarily met in the usual methods of analysis. Reproductions of these are given and the optical properties of each acid.

Determinative Table for the Amino Acids.—The crystalline material is immersed in the liquid indicated and then examined under the microscope with the polariser in place and the diaphragm partly closed. The results shown in the following table were recorded:

Immersion. Liquid. (n)	Remarks.	Amino Acid.
1·515 (n_α)	$n_\beta=1{\cdot}575$ also common	serine
1·535 (n_β)	On plates not extinguishing sharply	leucine
1·565 (n_γ)	Lengthwise on rods	valine
1·575 (n_γ)	,, ,, ,,	alanine
1·605 (n_β)	Fragments show orange and blue bands of colour	glutamic acid
1·615 (n_β)	Fragments remain practically bright with crossed nicols	glycocoll
1·630 (n_γ)	On many fragments with sharp extinction	aspartic acid
1·635 (n_γ)	—— ——	tryptophane
1·675 (n_γ)	$n_\alpha=1{\cdot}600$ also common	phenylalanine
1·680 (n_γ)	Lengthwise on needles	tyrosine
1·700 (n_γ)	Crystals show orange and blue bands	cystine

P. H. P.

Inorganic Analysis.

New Absorbent for Oxygen in Gas Analysis. L. F. Fiesher. (*J. Amer. Chem. Soc.*, 1924, 46, 2638–2647.)—An alkaline solution of sodium hydrosulphite containing sodium anthraquinone-β-sulphonate is recommended as an absorbent for oxygen; it forms a deep red coloured solution which becomes brown when exhausted. The advantages over pyrogallol are that under no conditions can it give rise to carbon monoxide, and it compares favourably with sodium hyposulphite alone, in that it not only absorbs more rapidly and completely, but its rate of absorption does not diminish until it is quite exhausted. The sodium anthraquinone-β-sulphonate acts solely as a catalyst. For a pipette designed for shaking, 16 grms. of hydrosulphite ($Na_2S_2O_4$, 86·7 per cent.), 6·6 grms. of sodium hydroxide, and 2 grms. of the sulphonate are dissolved in 100 c.c. of water, and for the bubbling type of pipette the corresponding quantities are 16, 13·3, and 4 grms., respectively. The rate of absorption is just a little less than that of alkaline pyrogallol, but the results are satisfactory. H. E. C.

Gasometric Determinations by means of Combustion with Copper Oxide. J. Svéda. (*Chem. News*, 1925, 130, 1.)—The determination of combustible gases (methane, hydrogen, carbon monoxide) by burning in contact with copper oxide has hitherto been carried out only in the presence of a current providing an excess of air. The author describes a method by which the oxygen necessary is derived solely from a mixture consisting of three parts of the cupric oxide with one part of ceric oxide, the presence of which prevents the cupric oxide from becoming too compact after repeated use. (It is also said to act as a catalyser in the combustion.) The mixed oxides are contained in a small porous crucible in the eudiometer and can be heated electrically to the desired temperature (280–290° C.). The porous crucible consists essentially of a conical spiral of platinum wire covered with Marquardt paste, which must then be very carefully dried and heated before use. The copper oxide is packed into the porous vessel covered with Marquardt's paste, carefully dried, and the whole placed in a glass tube, 6 mm. in diameter. The platinum leads are fused through the glass, and the tube bent to a U-shape. It is then graduated by etching with hydrofluoric acid and calibrated with mercury. In experiments with pure hydrogen the eudiometer was filled with a definite volume of the gas and transferred to a glass cylinder filled with mercury. The copper-oxide vessel was introduced into the gas and heated to 280–290° C., when a visible contraction took place. The heating was continued for half-an-hour, and the residual gas was dried by the introduction of a bead of metaphosphoric acid or magnesium perchlorate, which was left in it for several hours. Experimental mixtures of pure hydrogen and specially purified nitrogen were treated by this method and shown to give very satisfactory results. In experimenting with carbon monoxide (prepared by the action of sulphuric acid on oxalic acid, and collected over mercury after eliminating air, carbon dioxide and moisture) the same procedure was followed, but the results were quite unsatisfactory. The discrepancy was attributed to differences in the adsorption power of cupric oxide for carbon dioxide and nitrogen. Furthermore, the air occluded by the copper oxide is very difficult to remove. These adsorption errors have been overcome, as will be described in Part II. R. F. I.

Identification of Metals as Double Halogenides with Pyridine, Antipyrine, Pyrazole or Aniline. I. M. Kolthoff and H. Hamer. (*Pharm. Weekblad*, 1924, 1222–1227.)—It has been found that copper, cadmium, zinc and nickel combine with a cyanide and pyridine to give precipitates with the general formula $M.(CNS)_2Py_2$. The sensitiveness of the reaction depends upon the amounts of reagents used, the best proportions being 5 c.c. of metallic solution, 0·5 c.c. of 20 per cent. potassium or ammonium thiocyanate, and 1 c.c. of 10 per cent. pyridine.

This reaction is not specific, as all metals give it, but if a chloride, bromide or iodide be substituted for the thiocyanate, fewer metals are precipitated. In this respect the bromide test is the most sensitive, and 5 mgrms. of zinc or cadmium per litre can be determined. Other weak bases have been substituted for pyridine, but positive results were obtained only with antipyrine and pyrazole. The test, with the use of antipyrine and a thiocyanate can be used to detect 1 per cent. of zinc in the presence of cadmium. By the use of pyrazole and a thiocyanate one mgrm. of copper can be detected, a violet coloration being produced. Microchemically, zinc and cadmium may be detected by the crystalline form of their double halogenide salts when the pyridine and bromide reagents are used. Iron and aluminium interfere with all these tests and must be removed before they are applied. W. S. S.

Copper as a Reducing Agent in the Determination of Iron. J. M. Hendel. (*Ind. Eng. Chem.*, 1924, **16**, 951.)—Boiling ferric sulphate solution may be reduced completely by means of copper gauze immersed in it; 64 sq. cm. of the gauze will reduce 0·150 grm. of iron in fifteen minutes. After reduction, the solution is cooled, aerated for three to five minutes to oxidise cuprous and titanous salts, and the ferrous salt is then titrated with permanganate solution. The acidity of the solution during the reduction process should be 0·1 *N*, and increased to normal before the aeration and titration. The aeration, which should be at the rate of 1 litre of air per five minutes, does not oxidise the ferrous salt. It is advisable to filter the air through cotton wool before use. W. P. S.

Analysis of Germanite (Determination of Germanium, Arsenic, Gallium, and Molybdenum). F. W. Kriesel. (*Chem. Zeit.*, 1924, **48**, 961–963.)—Germanite, discovered in S.W. Africa, is richer in germanium and gallium than any other known mineral. Its composition is: Cu 45·39, Pb 0·66, Zn 2·58, Fe 4·56, Ge 8·70, S 30·65, As 4·13, Ga 0·76, SiO_2 0·226, WO_3 0·184, TiO_2 0·004, Mo 1·282, Mn 0·02, Ni 0·001, Co 0·013, Cd 0·071, CaO 0·122, MgO 0·055, C 0·136, Ag+Au 0·005, total 99·549 per cent. *Determination of germanium.*—Two grms. of finely powdered mineral are digested overnight with nitric and sulphuric acids, and the former evaporated without fuming. The mass is dissolved by warming with 50 c.c. of water, and the solution rinsed into a distillation flask with 150 c.c. of strong hydrochloric acid; any germanium dioxide adhering to the beaker is dissolved in ammonium sulphide and added to the solution. The flask is connected with a spiral cooler, and the latter with a receiver, cooled in ice, and containing 20 c.c. of dilute hydrochloric acid. A slow current of chlorine is passed through the apparatus during the distillation, which is interrupted when two-thirds of the liquid have passed over. About 90 per cent. of the germanium distil with the first 10 c.c. The chlorine is expelled from the apparatus by carbon dioxide, and the distillate treated for half an hour with hydrogen sulphide. A heavy, granular, pure white precipitate of germanium disulphide soon forms; the strong acidity required for its quantitative precipitation is ensured by the above procedure. After settling, the precipitate is collected, dissolved in a little ammonium sulphide, and the resulting solution (100 c.c.) acidified with 50 c.c. of sulphuric acid (1:1) and treated with hydrogen sulphide for 10 minutes. The voluminous precipitate is filtered off and washed three times with 20 per cent. sulphuric acid saturated with hydrogen sulphide, followed by three washings with absolute alcohol. The re-precipitation ensures complete absence of chlorine, which would cause low results by volatilisation. The washed precipitate is rinsed into a platinum basin, the paper rinsed down with ammonia, the liquid evaporated, the residue cautiously moistened with strong nitric acid, again dried, and ignited gently to expel sulphuric acid; these operations are repeated. The ignited residue is moistened with ammonia, dried, and ignited; this treatment is repeated till the weight of the dioxide is constant. It is a heavy, soft powder of snow-white colour. *Arsenic.*—The residual liquid in the distillation flask is acidified with 100 c.c. of strong hydrochloric acid, and distilled in a current of sulphur dioxide to as small a bulk as possible; the distillation is repeated with another 100 c.c. of acid, when all the arsenic will be in the distillate. This is treated, while being cooled, with solid permanganate until the brown colour is permanent; the latter is discharged by sodium hypophosphite, and the arsenic precipitated with hydrogen sulphide. *Gallium.*—Fifty grms. are dissolved, and the nitric acid removed, as before. The mass is dissolved in 300 c.c. of water by warming, and the lead sulphate and germanium dioxide filtered off after cooling. The filtrate, diluted to 500 c.c., is electrolysed for 24 hours at 2 amp. between gauze electrodes. The copper deposit becomes black and spongy (arsenic, molybdenum) towards the end. The electrolyte is filtered into a beaker and evaporated to fumes after addition of 10 c.c. of nitric acid. The mass is taken up in 400 c.c. of water, and the solution precipitated exhaustively with hydrogen sulphide to eliminate the remaining germanium, arsenic, and molybdenum. The precipitate is collected and washed with acidulated water; the washings are caught separately, evaporated to small bulk, strongly acidified, precipitated with hydrogen sulphide, and filtered into the main filtrate (germanium disulphide is soluble in water). The filtrate is boiled for the removal of hydrogen sulphide, the iron oxidised with bromine, and the iron, aluminium, and gallium obtained together by double basic acetate precipitation. The precipitate is dissolved in a minimum of sulphuric acid, the iron reduced by hydrogen sulphide, and the sulphur filtered off. The hydrogen sulphide is boiled off, the liquid cooled and treated with sodium carbonate to faint acid reaction, then boiled a few minutes with excess of a suspension of precipitated cuprous oxide: gallium and aluminium are precipitated. The precipitate is collected and washed with hot water, dissolved in dilute nitric acid, and the solution evaporated to fumes with sulphuric acid. The copper is removed by electrolysis; the electrolyte is again treated with hydrogen sulphide, etc., and the whole cuprous oxide separation repeated to remove the last of the iron, the copper being once more deposited electrolytically. The liquid, which now contains gallium and aluminium, is made faintly ammoniacal and boiled until its reaction is feebly acid. The voluminous precipitate is collected and washed with hot water. The following separation from aluminium is more convenient than the ferrocyanide method;

the precipitate is dissolved in dilute sulphuric acid, the acid is almost completely neutralised with sodium carbonate, and the solution diluted to 500 c.c. after addition of 5 grms. of sodium acetate. A suitable quantity of sodium arsenite is now added (equivalent to twice as much arsenic as the gallium present), and the cold liquid saturated with hydrogen sulphide; gallium is quantitatively precipitated in presence of arsenic. The precipitate must be washed in the cold, otherwise aluminium may be precipitated. The filtrate is tested for quantitative precipitation of the gallium by the ferrocyanide reaction, after being strongly acidified with hydrochloric acid: no precipitate should form. The sulphide precipitate is dissolved in *aqua regia* and the solution evaporated to fumes with sulphuric acid. Any silica is filtered off after dilution and the arsenic precipitated as sulphide. The filtrate is boiled to expel hydrogen sulphide, and the gallium precipitated with ammonia. The precipitate is filtered off, dissolved in nitric acid, and the precipitation repeated (removal of chloride). The precipitate is ignited and weighed as Ga_2O_3. *Molybdenum.*—Twenty-five grms. are digested for some time with 100 c.c. of nitric acid. The solution is diluted, filtered, treated with one grm. of iron as nitrate, neutralised first with strong, then with dilute, sodium carbonate solution to a permanent faint turbidity, diluted to 1500 c.c., and kept hot for 1 to 2 hours. If the quantity of iron is sufficient, the precipitate contains all the arsenic, molybdenum, bismuth, selenium, and tellurium. The precipitate is washed free from copper. A second precipitation, after addition of 0·5 grm. of iron, is carried out in the filtrate. The combined iron precipitates are dissolved in hydrochloric acid, and the solution precipitated exhaustively with hydrogen sulphide. The sulphides are treated with sodium sulphide; the resulting solution is acidified with hydrochloric acid, the precipitate coagulated by warming, collected, and dissolved in hydrochloric acid and potassium chlorate. The chlorine is expelled and the sulphur filtered off; it is burned off at low temperature, the fixed residue treated with hydrochloric acid and chlorate, and the solution added to the bulk. Selenium and tellurium are eliminated by sulphur dioxide. The filtrate contains molybdenum and arsenic; it is evaporated almost to dryness, the residue is taken up with 100 c.c. of strong hydrochloric acid, the solution saturated with sulphur dioxide, and again evaporated as before: arsenic is completely volatilised. The residue is evaporated with 10 c.c. of nitric acid almost to dryness, the mass taken up with hot water and a little nitric acid, and the solution made ammoniacal and filtered. The filtrate is acidified with acetic acid, and the molybdenum precipitated as lead molybdate. W. R. S.

New Organic Reagent for the Detection of Nitrates and Perchlorates. C. S. Marvel and V. du Vigneaud. (*J. Amer. Chem. Soc.*, **1924, 46,** 2661–2663.)—The new reagent is α-phenyl-β-diethylaminoethyl-*p*-nitrobenzoate, which forms insoluble salts with nitric and perchloric acids. Although it is not quite so sensitive as nitron or a di-(α-naphthylmethyl)-amine, it has the advantage of being specific. When applied in a 30 per cent. solution in dilute hydrochloric acid, nitrates or perchlorates give a white precipitate which is sensitive down

to 0·005 *N* and 0·0025 *N* respectively. Other anions, including nitrites and chlorates, do not interfere, but iodides, oxalates, thiocyanates and dichromates do.
H. E. C.

Determination of Phosphorus in Phosphor Bronzes. L. Lindemann. (*Ind. Eng. Chem.*, 1924, **16**, 916.)—The method depends on the complete oxidation of the phosphides, prevention of the hydrolysis of the tin, and precipitation of the phosphoric acid as ammonium phosphomolybdate. About 0·2 grm. of cast phosphor bronze, or 0·5 grm. of malleable bronze, is dissolved in a mixture of 25 c.c. of hydrochloric acid (sp. gr. 1·10) and 5 c.c. of nitric acid; the mixture may be heated slightly at first, but the reaction must not be allowed to proceed too violently. The solution is then heated nearly to boiling for ten minutes to expel chlorine, diluted with 50 c.c. of cold water, 10 c.c. of dilute ammonia (1:1) and 50 c.c. of molybdic acid reagent are added, the precipitate is collected, and the phosphorus estimated alkalimetrically.
W. P. S.

Applications of the Method of Electrolysis with the Dropping Mercury Cathode. J. Heyrovsky. (*Compt. rend.*, 1924, **179**, 1267–1268.)—Applications of the dropping mercury cathode described by the author (*Phil. Mag.*, 1923, **45**, 303; abst. *J. Chem. Soc.*, 1923, **124** ii, 119), are given, and it is shown that the method is useful in the investigation of solutions having very low ionic concentrations of the metals. Thus alkaline zincates and plumbates can be examined. It may also be applied to non-aqueous solutions of organic or inorganic substances. The difference between the decomposition potential π_n at the dropping cathode and the normal electrolytic potential E.P., multiplied by a factor F, expresses the affinity of amalgamation A of an equivalent of the metal; this represents the energy set free by the combination of the metal with the mercury. Numerical data are given in the paper.
H. E. C.

Electrometric Titration of Hydrazine and its Salts. E. C. Gilbert. (*J. Amer. Chem. Soc.*, 1924, **46**, 2648–2655.)—Although the curve representing the titration of hydrazine with acid in the presence of an indicator such as methyl orange shows only one point of inflexion, indicating a mono-acid base slightly weaker than ammonia, electrometric titration reveals a second point of inflexion. This indicates a di-acid base in which the second stage in the ionisation corresponds with the formation of an unionised base N_2H_5OH or $N_2H_6(OH)_2$. The value of K_2 is less than 1×10^{-12}. Hydrazine may also be titrated electrometrically or stoichiometrically with either iodine or bromate solutions; the results of the two methods agree well. For the electrometric titration a 0·1 *N* calomel cell is used in the ordinary way.
H. E. C.

Electrometric Titration of Antimony and Tin by Potassium Dichromate. M. H. Fleysher. (*J. Amer. Chem. Soc.*, 1924, **46**, 2725–2727.)—In the ordinary titration of trivalent antimony or divalent tin by potassium dichromate there is some difficulty with the end-point. This may be overcome by the use of the electrometric method; an ordinary normal calomel cell and platinum electrode is employed, and there is a well-marked change in voltage of about 100 mv. at the end of the titration. It is advisable to eliminate atmospheric oxidation by replacing the air by carbon dioxide or hydrogen during the titration. Antimonious chloride can be determined in the presence of stannous chloride by the addition of mercuric chloride, which oxidises the tin, but has no other influence on the course of the titration. Arsenious chloride can be estimated in an analogous manner.

H. E. C.

Physical Methods, Apparatus, etc.

The Ultra-Centrifuge, A New Instrument for the Determination of the Size of Particles in Amicroscopic Colloids. T. Svedberg and H. Rinde. (*J. Amer. Chem. Soc.*, 1924, **46**, 2677–2693.)—An instrument has been devised for the measurement of the size and distribution of the particles in amicroscopic colloids about which but little is at present known. It consists essentially of a high speed centrifuge in which special precautions are taken to eliminate vibration and any small changes of temperature which give rise to convection currents. The colloid is centrifuged in this instrument, and the rate of movement of the boundary between the particles and the colourless liquid is observed either by means of a telescope or photographically, then the size of the particles and their distribution is calculated by means of equations developed from Stokes's formula as modified by Svedberg and Nichols. As an example, the size and distribution of the gold particles in gold sols are shown, having radii varying in different sols from 2·3 to 11·6 $\mu\mu$. The determinations give values from 11 to 38 per cent. higher than those given by Zsigmondy's nuclear method. H. E. C.

Method for the Determination of the Distribution of Size of Particles in Emulsions. E. O. Kraemer and A. J. Stamm. (*J. Amer. Chem. Soc.*, 1924, **46**, 2709–2718.)—When an emulsion in which the disperse phase tends to rise under the influence of gravity is placed in a wide vertical tube with an upturned capillary side-tube containing the dispersion medium, as the disperse phase rises there is a corresponding change of density which causes a change in the height of the liquid in the capillary. An instrument based on this principle has been designed, so that by observing the changes in position of the liquid in the capillary tube under suitable conditions the corresponding changes in pressure in the emulsion in the vertical tube, caused by the rising of the particles, can be calculated with the help of the equations which have been elaborated. From these expressions the distribution and the size of the particles can also be calculated. Emulsions of benzene and water, stabilised by potassium oleate and palmitate, have been investigated by this method, and, among other results, some confirmation of the wedge theory of emulsions is afforded by the fact that potassium oleate tends to give an emulsion with larger drops than does potassium palmitate.

H. E. C.

Continuous Conductivity Method of Measuring Small Concentrations of Chlorine in Air. T. B. Hine. (*Ind. Eng. Chem.*, 1924, **16**, 952–953.)—The air containing small amounts of chlorine is scrubbed continuously and rapidly with distilled water which is then allowed to flow through a conductivity cell. The electrical resistance of the cell is a function of the chlorine concentration of the air and serves as a measure of this concentration. A glass scrubber operating on the principle of the Ceco sprayer is recommended; in this apparatus the water falling on the centre of a rapidly revolving horizontal disc is thrown off to the sides in a thin sheet through which the air to be scrubbed must pass. The volume of liquid in the scrubber at any time is small, and there is practically no resistance to gas flow. The apparatus is standardised with air containing known amounts of chlorine. W. P. S.

Economic Anode for rapid Electrolysis. A. Lassieur. (*Bull. Soc. Chim.*, 1924, **35**, 1530–1532.)—The rotating anode consists of a glass rod bent in such a manner that its revolution will rotate the electrolyte. A thin platinum wire (diam. 0·3 mm.) is wound round the rod and communicates at the upper end with the anode terminal. In addition to the small quantity of platinum required, this form of anode presents the advantage of a small conducting surface, on which oxidation reactions are reduced to a minimum. W. R. S.

Reviews.

QUANTITATIVE ORGANIC MICRO-ANALYSIS. By F. PREGL. Second Edition. Translated by E. FYLEMAN. Pp. xv.+190, with 42 illustrations. London: J. & A. Churchill. 1924. Price 12s. 6d. net.

The translator of this volume has rendered a valuable service to British chemists by providing what appears to be the first text-book on this subject in the English language. Although much work of this nature has been carried out in America and in Germany, comparatively little advance in the application of micro-methods has been made in this country, and the publication of this volume should provide a stimulus to the adoption of procedures which are invaluable in many branches of organic investigation. The author was one of the pioneers in the application of micro-methods to quantitative organic analysis and has spent several years in the perfection of the methods described in this volume, these having been thoroughly tested by his colleagues and students and subsequently applied to researches in many branches of organic chemistry.

The subject matter consists of detailed descriptions for the setting up and manipulation of the necessary apparatus and methods for the determination of the usual elements and radicals in organic compounds, together with the micro-electrolytic estimation of copper and the boiling-point determination of molecular weights with the use of 1·5 c.c. of the solvent and 7 to 10 mgrm. of the substance under examination. The delicacy and utility of the methods advocated will be appreciated when it is realised that a complete and accurate determination of carbon and hydrogen may be carried out with 2 mgrms. of material in about 45 minutes, and that a determination of copper in 20 to 25 grms. of preserved vegetables by the micro-electrolytic method may be completed in a little over one hour, with an accuracy within 1 per cent. of the metal present. Such methods are obviously only possible with a balance of precision, and a modified assay balance constructed by Kuhlmann to the author's specification is described; this is capable of turning to 0·001 mgrm. with a maximum load of 20 grms.

Not only are the manipulations described given with a wealth of essential detail infrequently met with in English books, but the author has also included in several instances the different stages reached in the development of the methods finally adopted. This feature will serve as a valuable example of manipulative evolution to the student, for whom a course of micro-analysis would provide an admirable training, but will be somewhat of a hindrance to the busy analyst. The translator has endeavoured to render his interpretation of Dr. Pregl's work as literal as was consistently possible, and the phrasing is consequently in a few instances somewhat quaint, and, in addition, one notices frequent references to different materials used being "of groat to hempseed size." The text of this volume is practically free from error, but a few references to other parts of the book are incomplete, since the number of the page is omitted, and on page 87 "grains" is printed instead of "grams," and cadmium on page 158 is rendered "cadium."

After studying the text of this excellent work one experiences a feeling of disappointment on turning to the index, for this is by no means on a level with the rest of the book. Many important sections and items in the text are entirely omitted, and, on the other hand, several references are given to pages upon which a substance or piece of apparatus is only incidentally referred to and their inclusion in the index is therefore useless. Thus under the index heading of "Combustion tube," we find on page 42 "connecting it with the neck of the combustion tube"; on page 65 "the rubber stopper at the mouth of the combustion tube" and so on. A further omission from the index is that of many necessary sub-titles, and it is somewhat bewildering to look up one title such as "Hydrogen" and find fifteen references without any indication given of the particular use or determination to which each refers. Such a defect seriously detracts from the value of the book as a whole and should be remedied at the earliest opportunity.

The methods described are in every way excellent, and, after a brief training, the chemist will fully appreciate the great economy of time, space, reagents, and material under examination which are attained by their use. With the exception of the index, the volume is an admirable production, and the author, translator and publishers are to be congratulated upon their success in providing a volume which will be eagerly read, not only by those to whom micro-analysis is an essential, but also by the average chemist who will find much material scattered throughout the book that will be applicable in his own work. T. J. WARD.

A SYSTEMATIC COURSE OF QUALITATIVE CHEMICAL ANALYSIS OF INORGANIC AND ORGANIC SUBSTANCES. By HENRY W. SCHIMPF, M.D. Fourth Edition, revised by ALFRED I. CONE. Pp. ix.+201. New York: John Wiley & Sons, Inc.; London: Chapman & Hall, Ltd. Price 8s. 6d. net.

This book was written for American students of pharmacy and consequently, from an English point of view, suffers from bad spelling and queer phrases and a host of other infirmities which we in England have come to consider as inherent in many American publications. In addition, there are several misprints (p. 10, "metadihydrohybenzol" for "metadihydroxybenzol," and p. 172, line 25, "quinine" for "quinidine") and, of course, the few references given are to the U.S. Pharmacopœia.

The opening section of the book deals with general considerations and contains rather scanty accounts of such topics as the Ionic Theory and the Law of Mass Action.

The second part gives, without attempting either to be exhaustive or to depart far from the accepted routine, an account of the ordinary procedure for qualitative inorganic analysis. Each group is treated under four main headings:—(1) The special reactions of the metals in that group; (2) A table of these reactions in the form of a comparison chart; (3) The group separation; (4) Notes on the peculiarities of the group.

These four sections overlap, and hence much unnecessary repetition occurs, so that it is difficult for a student to get a clear view of the main features of group analysis. The acids, both organic and inorganic, are treated in the usual way, *i.e.* a list of the special reactions of each followed by a scheme for their detection.

The final section deals with organic compounds of pharmaceutical value. It opens with a scheme for the detection of carbon, hydrogen, nitrogen, chlorine, phosphorus and sulphur—and omits entirely the sodium fusion method. The major part of the chapter is taken up with the special reactions of compounds such as santonin, strychnine, etc. It may be noted that a new test for quinine is recommended. This test involves the use of Labarraque's solution.

On the whole, although there is nothing very new or very wrong in it, the book offers no great advantages to those studying pharmacy in England, and, an English book is perhaps preferable for those would-be pharmacists who hope to practise in England.

HAROLD TOMS.

THE EXTRA PHARMACOPŒIA OF MARTINDALE AND WESTCOTT. Revised by W. HARRISON MARTINDALE, Ph.D., and W. WYNN WESTCOTT, M.B., D.P.H. Eighteenth Edition in two volumes. Volume I. London: H. K. Lewis & Co., Ltd. 1924. Price 27s. 6d. net.

It is only four years since the last edition of this highly esteemed and indispensable compendium of medicines appeared, yet it has increased in bulk by about 50 pages, notwithstanding the numerous excisions that have been made by its revisers. For the chemist, as well as for the physician, the attractiveness of

"Martindale" will remain so long as it continues to be compiled on its present lines, because none of the articles is a mere statement of bald facts. The facts are there for every one to read and apprehend, but they are imbued with the imagination and the constructive spirit of the authors. How to write a preface is a question that chains most writers to the earth. The preface of this volume is an example of what can be done when courage, conviction and knowledge combine to guide the pen.

The monographs to which attention should be directed are many, and they thoroughly represent the rapid advances that are taking place in the field of therapeutics. Chemists will obtain a good idea of the return to favour of some of the metallic compounds. There is, perhaps, not so much importance to be attached, at the moment, to any increase in synthetic organic chemicals produced for medical purposes. On the other hand, many elaborate investigations have been made into the preparation and modes of action and methods of administration of colloidal metals, to which about twenty pages are given in the Extra Pharmacopœia. Basil Valentine seems to be in a fair way to have his belief in the medicinal virtues of antimony justified, if we may judge by the results which are being obtained from its use in protozoal infections. An excellent account of this work has been compressed into about a dozen pages. There is also given much information on the manufacture of the alkali-bismuthyl tartrates and the procedure to be adopted in the administration of them in syphilis; this is a matter in which considerable confusion prevailed, but Drs. Martindale and Westcott have succeeded in clearing up some of the mistakes as to the composition of these compounds.

Among other articles which have a claim for special mention are those on the alkaloidal periodides, the vaccines and antitoxins, potassium iodide and its use in goitre, cod-liver oil and its relation to the newer views on vitamins, and the preparations of chaulmoogra oil and their usefulness in the treatment of leprosy. For the chemist not the least useful feature of this work is the information that can be found as to the identity and composition of proprietary articles with more or less fancy names. A trial of the index for the tracing of these was very satisfactory; in fact, only one blank was drawn, and that was Nizin, which, by the way, was missing from the index of the previous edition.

It is a matter of supererogation to recommend what has already been designated as indispensable. The publishers' part has been accomplished most satisfactorily in every respect. WILLIAM KIRKBY.

THE CONCENTRATION OF SULPHURIC ACID. (Vol. III. Lunge Cumming Series.) By JOHN WILFRID PARKES, M.Sc., A.I.C. Pp. xii.+394. London: Gurney & Jackson. 1924. Price 31s. 6d. net.

The section dealing with this subject in the last edition has been completely revised and issued as a separate volume, and new matter has also been introduced.

The first two chapters give an outline of sulphuric acid concentration processes, and the properties of ordinary concentrated sulphuric acid. This latter chapter is extremely good and contains a large amount of most useful data, a great advantage being that only the most reliable information is given.

The third chapter describes concentration in lead pans, glass, platinum and other vessels. Various cascade plants are discussed, in which vitreosil and different acid-resisting alloys are used, and of these alloys a good description is given; the different types of plant are compared, with data on fuel consumption, etc.

In Chapter IV. concentration by various systems which make use of hot gases is very fully described. The Trepex, Kessler, Perrin, Duron, Gaillard, Gilchrist, and other plants are treated in detail. Much useful information is given as to fuel consumption and the working of the plant, more especially in connection with the Kessler and Gaillard plants.

A useful section taken from the Technical Records of Explosives supply works out in detail, among other things, the heat required for acid concentration.

The performance of the Cottrell and other electrical precipitators is dealt with in a chapter on the Concentration of Sulphuric Acid Vapours. The Calder high-speed scrubber is also an efficient plant, but the chief effect of either this or electrical methods is on suspended particles rather than on acid in the gaseous phase.

Chapter VII. discusses the recovery of acid from waste acid in petroleum refining, nitration processes, and in metal works. A fuller description of recovery from pickling and other acid waste liquors would have been of interest, as would also information on the use of film evaporators which have recently been used for this purpose, constructed in special lead alloys.

Chapters VIII., IX. and X. treat respectively, costs and efficiencies, transport of sulphuric acid, and statistics on its application.

Valuable information can be obtained from the Quinan method of costing used in H.M. Factories, and figures are given showing this in detail. The efficiency of the three general processes in use during the war is of interest, *i.e.*

Cascade	..	85–90 per cent.	Average Efficiency (1914–1918).
Kessler	..	91–93 „	„ „
Gaillard	..	96–97 „	„ „

The experience at Gretna, with waste acid from explosives, showed the Gaillard plant to be the most economical in working in all respects save fuel consumption. When dealing with chamber acid the Kessler plant is more flexible and less costly to install, but both were found superior to the Gilchrist plant.

This volume is well written and, like the previous edition, will be recognised as the standard work on the subject of sulphuric acid concentration. ERIC REAVELL.

THE MODERN SOAP AND DETERGENT INDUSTRY, INCLUDING GLYCEROL MANUFACTURE. Vol. I. THEORY AND PRACTICE OF SOAP-MAKING. By GEOFFREY MARTIN, D.Sc., F.I.C. Pp. 367+xii. London: Crosby, Lockwood & Son. 1924. Price 36s. net.

This is described as the first of three volumes which are to form a complete treatise on the Manufacture of Laundry, Toilet, Pharmaceutical, Textile, Abrasive, Scouring, and Powdered Soaps; also Detergent Compositions and Soap Substitutes of all kinds, and "the author hopes to justify the claim that no publication on soap so complete has appeared in any language."

The present volume deals with "the theory of soap, raw materials, calculation of charges, lay-out of soap factories, and the manufacture of ordinary household soaps by the usual processes." It is divided into seven sections, the first, on the Nature of Soap and Detergent action, being largely a reproduction of the very comprehensive article on the Colloidal Chemistry of Soap, by Prof. McBain in the Third Report on Colloid Chemistry, published by H.M. Stationery Office in 1920, while of the three following sections dealing with Raw Materials—Alkali and Alkali Salt; Oils and Fats; and Essential Oils and Perfumes—the two last appear to be abstracted almost verbatim from the author's works dealing with these two subjects, liberally interspersed with copious extracts, unacknowledged, from another text-book on the subject of Soap. In the section on Alkali, the causticisation of soda ash is very well described, but no mention is made of the fact that soap-makers are now taking up the manufacture of cement in order to dispose economically of the calcium carbonate obtained as a waste product. The author gives a very inadequate explanation of the "English" alkali degrees, and his definition of the American degrees as "the percentage of Na_2O present" is entirely wrong. The author's books on *Oils and Fats*, and *Essential Oils and Perfumes*, have already been reviewed in the pages of THE ANALYST (Nov. 1920, p. 432, and Dec. 1921, p. 527), so that it is unnecessary to discuss these sections at length, but it may be pointed out that the defects to which attention was then drawn, have not been remedied in any way in the present volume, and the reviewer would like to protest against the repetition of such statements as that the titre of a fat is "the melting point of the separated fatty acids," that "the refractive index of oils is usually determined at 60° C.," and that the melting point of an essential oil is determined by melting some of the solid oil, sucking it up a glass capillary tube, and attaching this to the bulb of a thermometer immersed in a bath of water, glycerin, or oil, which is then heated until the crystals in the tube melt and rise to the surface, this temperature being regarded as the m.pt. Further, coconut "oleine" and palm kernel "oleine" do not consist "mainly of free fatty acids," although anyone without special knowledge of the industry might think so in view of the final *e*.

These three sections which contain chapters on the analysis of the raw materials and should be of most interest to the analyst, are so full of errors, obsolete methods, and incomplete and misleading statements, as to be worse than useless in inexperienced hands, in spite of the fact that in the preface it is stated: "Very full methods of analysis of the various products are given, which it is hoped will enhance the value of the book to the analytical and works chemist."

The remaining sections, covering only some 173 pages—less than half the volume—are devoted to the manufacture of soap, Section V., from Free Fatty Acids, Section VI., by the Boiling Process, and Section VII., Household and Laundry Soaps. In Section V. it is difficult to understand the statement on p. 4 that "the Twitchell process has made possible the large scale saponification of fats for the production of crude glycerin free from salt," since "saponification glycerin," resulting from the saponification of fats in an autoclave with lime or magnesia, was on the market long before the introduction of the Twitchell process. The castor seed ferment process is referred to as the "Connstein process," although Sudborough, Watson and Varna have shown that Nicloux's method for the production of the active reagent is the most suitable for oil-splitting on a large scale, and a period of 6–8 hours is much too short for anything approaching complete hydrolysis, usually two to three days being required. Section VI. is very copiously illustrated with various forms of plant used, and proposed for use, in the soap industry, but it is largely made up of abstracts from patents and trade catalogues, without any practical guidance as to their merits, and the whole of this section, as well as the following one, while accurately reproducing what may be termed the text-book methods of making soap, is very scrappy. Section VII., which includes rosined soaps, genuine and filled mottled soaps, cold process soaps, and soaps from waste fats, is, again, largely a compilation of abstracts from official reports, bulletins, patents, and published formulæ, and contains much that is now out of date, which is not surprising in view of the fact that the author has made use of old editions of many works quoted and acknowledged (with dates), much later and more up-to-date editions of which are now available. It is from every point of view a disappointing book. W. H. SIMMONS.

ANTIQUITIES: THEIR RESTORATION AND PRESERVATION. By A. LUCAS, O.B.E., F.I.C. Pp. 135. London: Edward Arnold & Co. 1924. Price 6s. net.

In this little unpretentious book Mr. Lucas embodies some of the results of his long experience in dealing with treasures from the past. Few chemists can have had his opportunities not only of studying the composition of museum exhibits of every kind and ascertaining the effects of time and exposure upon it, but also of applying the knowledge so gained to the task of preserving a marvellous collection of objects unearthed after thousands of years from the forgotten sepulchre of an Egyptian king. Some account of the remarkable chemical questions that were presented in the course of that work would have been welcome, but perhaps this is not yet permissible.

In view of the fact that many, if not most, of the curators of museums have little chemical knowledge, full directions for the preparation and use of the various reagents recommended have been given in very simple terms, and the methods of manipulation described can be followed readily by anyone. Methods of mending, restoring, preserving, and, in some cases, of cleaning every description of antique object likely to be shown in a museum are arranged in a form that is easy for reference. Even such specialised work as the restoration of faded writing and the treatment of old wood and stone is fully described, and, above all, the over-zealous restorer of the antique is taught when to stay his hand.

Apart from its value to a particular class of readers, the book is of general interest to every chemist, and may be profitably consulted by him in dealing with some of the out-of-the-way problems that he is occasionally called upon to solve.

The publishers may be congratulated on their enterprise in making generally available an interesting book in an attractive form. EDITOR.

MARCH, 1925. Vol. L., No. 588.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

ANNUAL GENERAL MEETING AND ORDINARY MEETING, HELD FEBRUARY 4, 1925.

The Annual General Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, February 4th. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

The Hon. Treasurer presented the accounts of the Society for 1924, which were adopted, and votes of thanks were passed to the Hon. Treasurer and the Hon. Secretaries.

Messrs. Marreco, Houseman and Brandon, chartered accountants, were appointed auditors of the Society's accounts for 1925.

The President delivered his Annual Address. Dr. Dyer proposed that a hearty vote of thanks be accorded to the President for his address and for his services in the chair, and that his permission be asked to print the address in THE ANALYST. This was seconded by Mr. R. L. Collett, and the motion was carried.

The following were elected as Officers and Council for the ensuing year:—

President.—G. Rudd Thompson.

Past-Presidents serving on the Council (limited by the Society's Articles of Association to 8 in number).—Leonard Archbutt, A. Chaston Chapman, Bernard Dyer, P. A. Ellis Richards, Alfred Smetham, E. W. Voelcker, and J. Augustus Voelcker.

Vice-Presidents.—S. F. Burford, E. M. Hawkins and A. More.

Hon. Treasurer.—Edward Hinks.

Hon. Secretary.—E. Richards Bolton.

Assistant Hon. Secretary.—R. G. Pelly.

Other Members of Council.—F. W. F. Arnaud, E. T. Brewis, R. T. Colgate, R. L. Collett, J. C. Drummond, C. A. Hill, L. H. Lampitt, T. Macara, S. E. Melling, J. Sorley, A. R. Tankard, and J. White.

An Ordinary Meeting followed the Annual Meeting, the re-elected President, Mr. G. Rudd Thompson, being in the chair.

Certificates were read for the first time in favour of:—Messrs. Douglas James Talbot Bagnall, A.C.G.F.C., A.I.C., Reginald Henry Coysh, M.Sc., A.I.C., George Van Barneveld Gilmour, B.Sc., A.R.C.Sc.I., A.I.C., Percy May, D.Sc., F.I.C., John Parry, Wilfrid Smith, B.Sc., A.I.C., and Winifred Wright, B.Sc., A.I.C.

A Certificate was read for the second time in favour of Mr. Thomas Mann.

The following were elected Members of the Society:—Messrs. Arthur Chapman Barnes, B.Sc., F.I.C., John Jacob Fox, D.Sc. (Lond.), F.I.C., Thomas Congalton Hay, and Harold Rupert Jensen, M.Sc. (Liv.), A.I.C.

The following papers were read:—"Cinchonine as a Tannin Precipitant, with Special Reference to the Analysis of Cutch and Gambier," by David Hooper, LL.D., F.I.C. (work carried out under the Analytical Investigation Scheme); "The Examination of Charred Documents," by C. Ainsworth Mitchell, M.A., F.I.C.; and "The Absorption of Carbon Monoxide in Gas Analysis," by H. R. Ambler, B.Sc., A.I.C.

Annual Address of the Retiring President.

On my retirement from office as your President it becomes my duty to render an account of my stewardship to you for the year 1924, and to give you at the same time a general resumé of what we as a Council have done and how we as a Society have fared during the period.

I think I can safely say that as a Society we are flourishing; we have been able to welcome some 45 new members, and there has not been a single meeting of the Society without a list of additions to our membership roll having been made.

We now stand with 495 members: 3 resignations have been received and 3 members have been removed: two for failure to pay subscriptions and one for having committed what in the opinion of the Council were acts "derogatory to the interests of the profession of analytical chemistry."

It is quite needless for me to remark that a society such as ours can only be carried on in a satisfactory manner by a rigid adherence to our constitution, by-laws and regulations, but I assure you the step was only taken after very mature and most careful consideration of the points at issue, and having full regard to the upholding of the dignity and traditions of our Society.

I regret, however, to report that we have lost 12 members by death, including Sir G. T. Beilby, Sir Charles Cameron and Sir J. J. Dobbie from the ranks of our Honorary Members, whilst of our Ordinary Members, Messrs. G. H. Allibon, M. K. Bamber, F. J. Bolt, L. Briant, C. B. Gall, L. Gowing Scopes, Otto Hehner, R. Hellon, and E. Richards have left us for the Great Beyond, and the Society is poorer for their loss.

e due to the production of an acid caused by the fermentation of sugars. tion allowed to stand a day or so before precipitation usually gave a eld of tannate than the fresh solution, and very dilute acetic acid cided solvent action on this compound. It is a disadvantage that there iformity in the composition of the cinchonine tannates, but eliminating eptionally low figure for nitrogen in mangrove tannate, the average of 3·4 3 will give the factor 0·6 to obtain the amount of tannin when extreme cy is not required.

IFFERENTIATION OF CATECHIN AND TANNIN.—The greatest disparity in vo methods is observed in the figures for tannin in cube gambier and the heart of *Acacia catechu.* This is due to the presence of catechin, which is absorbed large extent by hide powder, but is not affected by cinchonine sulphate. sample of cube gambier, for instance, contained 30 per cent. of crystalline iin: the hide powder evidently removed the whole of this principle together h the tannin. The catechu wood contained 4·8 per cent. of catechin, of which hide powder absorbed three quarters. Comparative experiments made by ating solutions of pure tannin mixed with weighed quantities of catechin emonstrated that the weight of tannate was practically the same as when no techin was present. A solution of gallotannin gave precipitates with cinchonine representing 69·9 per cent. of tannin. A solution of the same strength, with the addition of 1 grm. of acacatechin per litre, gave precipitates showing the presence of 70·5 per cent. of tannin.

ANALYSES OF CUTCH AND GAMBIER.—These conclusions point to the great alue that may be found in the cinchonine method in discriminating between techin and tannin in the analysis of cutch and gambier. The method was ployed in determining the tannin in the following representative samples of dian catechus or cutches, kindly forwarded by Dr. H. H. Mann, Director of iculture, Bombay.

(1) Catechu from Cawnpore, U.P. Re 1½ per lb.; (2) Catechu from Surat, rat, Re 1¼ per lb.; (3) Catechu in squares, Re 1 per lb.; (4) Catechu from Shirval, product, generally used for dyeing, Re 1 per lb.; (5) Catechu in small square als, Re 1 per lb.; (6) Catechu powder from Burma for dyeing, 8 as. per lb.; atechu from Burma, blocks for dyeing, 8 as. per lb.

	1 Per Cent.	2 Per Cent.	3 Per Cent.	4 Per Cent.	5 Per Cent.	6 Per Cent.	7 Per Cent.
ater	11·2	7·0	2·8	10·0	9·2	7·0	10·0
echin	43·4	10·6	1·0	13·8	24·2	12·4	15·0
nnin by cinchonine	16·6	4·5	3·0	44·0	23·0	17·1	44·5
s non-tannins	13·6	5·1	1·8	20·6	21·0	16·9	24·9
anic insol.	5·6	25·8	13·4	9·6	8·2	13·4	3·3
	9·6	47·0	78·0	2·0	14·4	33·2	2·3
	100·0	100·0	100·0	100·0	100·0	100·0	100·0

The catechin was separated and determined by crystallisation from a solution of the alcoholic extract, the mother liquors being evaporated, catechin separated as long as any crystals formed. This method was use author in finding the percentage of catechin in a large number of samples examined in India (*Agricultural Ledger*, No. 3, 1906). The figures in the instance were confirmed by evaporating and separating the crystals fro aqueous solution of the ethyl acetate extracts.

There appears to be no definite ratio between catechin and tannin in commercial samples of cutch. In 1 and 2 the catechin is more than doub amount of tannin, and the proportion is reversed in 4 and 7; in No. 5 the cat and tannin are nearly equally balanced. The results of the hide powder me are not given in the above table since they include catechin. It must be conc that the analysis showing the catechin separated in a crystalline condition, and tannin determined by cinchonine, shows the composition of cutch in a better v than the usual method of analysis.

These experiments indicate that although cinchonine sulphate is only fairly good quantitative precipitant of tannin in certain tanning materials, may have special value in determining tannin in the presence of catechin.

The author's thanks are due to Dr. Nierenstein for the interest he has shov in the work and the advice he has given.

BIOCHEMICAL LABORATORY,
UNIVERSITY OF BRISTOL.

DISCUSSION.

Mr. CHASTON CHAPMAN writes: "It is, of course, very gratifying to me find that my cinchonine method has given such good results in Dr. Hoope hands. That it is specially useful in the analysis of certain natural produ and commercial extracts containing both tannin and catechin I have known a considerable time, and I have, on several occasions, employed it in the examinat of catechu and similar materials. In such products the results for tannin ar course, much lower than by the hide powder process, since the cinchonine met gives the true tannin, whereas the hide powder gives the combined percen of all materials removable by that reagent.

It is interesting to observe that the nitrogen percentage which I four the cinchonine lupulo-tannate is very nearly that found by Dr. Hooper i corresponding compound with gallotannic acid, namely, 4·3 per cent. It that a study of the cinchonine compounds of the various so-called tannic might help materially to an understanding of the true nature of those substa

On the Absorption of Carbon Monoxide.

PART I.

A Critical Comparison of some Methods Employed in Gas Analysis.

By H. R. AMBLER, B.Sc., A.I.C.

(*Read at the Meeting, February 4, 1925.*)

THE absorption of carbon monoxide by cuprous chloride is frequently found to be not strictly quantitative. In this paper are investigated:

(A) The conditions governing the absorption of carbon monoxide by cuprous chloride. (*a*) Dissolved in hydrochloric acid; (*b*) Dissolved in ammonia.

(B) The use of a suspension of cuprous oxide in sulphuric acid containing β-naphthol, as an alternative and an improvement.

Attention has been directed particularly to:—(1) Completeness of absorption; (2) rate of absorption; (3) the effect, on the absorptive power, of carbon monoxide previously absorbed.

The apparatus used was of the Sodeau (constant pressure) type. The experiments were all conducted at atmospheric temperature.

ACID SOLUTION OF CUPROUS CHLORIDE.—This solution is prepared by dissolving 30 grms. of cuprous chloride in a mixture of 50 c.c. of water and 150 c.c. of concentrated hydrochloric acid. The solution is then kept in an air-tight bottle with a spiral of copper wire until the brown colour, due to cupric chloride, has disappeared.[*]

It was found that this solution did not absorb carbon monoxide completely. For instance, gas samples known to contain between 0·5 and 0·8 per cent. of methane[†] gave apparent percentages as high as 1·3, even when the gas had been shaken successively in three absorption pipettes, each containing fresh solution.

An artificial mixture of nitrogen, carbon monoxide and hydrogen (containing no methane) was analysed, with the following results:

	c.c.	c.c.
Volume of original gas (c.c.)	42·2	44·85
Volume after shaking with (old) solution No. 1	22·5	24·7
Volume after shaking, solution No. 2 (had previously absorbed about 4 c.c.)	21·9	23·5
Volume after shaking, solution No. 3 (fresh)	21·8	23·35
Volume after addition of oxygen	30·6	32·50
Volume after explosion	9·7	10·30
ol me after shaking with potash	9·35	10·00
ol e of carbon monoxide left unabsorbed	0·35	0·30

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[*] If cupric chloride is present the solution is not markedly less efficient, but cannot be a contact with mercury in the absorption pipettes.

[†] Methane is determined by measuring the carbon dioxide produced by its combustion, is carried out (together with that of hydrogen) after the removal of carbon monoxide. rb monoxide left unabsorbed will, of course, be determined as methane.

Rate of absorption and vapour-pressure of acid.—It is often stated that it is necessary to leave the gas in contact with the solution for as long as 10 minutes. It appears, however, that any carbon monoxide left unabsorbed after one minute's shaking will not be absorbed after any further contact with the same solution.

This is shown in the following analysis:

	c.c.	c.c.
Volume of gas after removal of CO_2 and oxygen	28·55	33·70
Volume of gas after shaking ½ minute with cuprous chloride	15·60	12·85
Volume after standing 20 minutes with cuprous chloride	15·60	—
Volume after standing 30 minutes with cuprous chloride	—	12·90

The slight increase in volume in the second case is probably due to increase in the vapour pressure of the hydrochloric acid; the volume reading, however, is not accurate, to much closer than 0·05 c.c.

For practical purposes this vapour pressure is negligible, as, *e.g.* in the following results:

	(1) c.c.	(2) c.c.	(3) c.c.
Volume after shaking with cuprous chloride	19·95	15·55	19·80
Volume after shaking with water	19·90	15·50	19·80

It is unnecessary, therefore, to wash the gas with water after acid cuprous chloride, especially as the errors due to incomplete absorption are of a much higher order than this vapour pressure effect.

AMMONIACAL SOLUTION OF CUPROUS CHLORIDE.—The solution is prepared by passing ammonia gas through a suspension of 20 grms. of cuprous chloride in 150 c.c. of water, in absence of air. The solution prepared under ordinary conditions, either from stock cuprous chloride or from the fully reduced precipitated compound is of a fairly deep blue colour, becoming lighter as it takes up carbon monoxide, until it becomes almost colourless. This loss of colour is a useful indication that the solution is becoming exhausted, as at about this point it begins to show a loss in efficiency. It is to be noted that if, in the preparation, care is not taken to exclude air, a solution is obtained of the same colour and showing the same colour change with use, but which is much less efficient.

Ammoniacal cuprous chloride, even when considerably oxidised, can be kept in contact with mercury indefinitely. It was found to be a considerable improvement on the acid solution, as is seen in the following results:

	c.c.
Volume of gas after shaking successively with three fresh acid CuCl solutions ..	15·50
Volume after shaking with water	15·50
Volume after shaking with ammoniacal cuprous chloride	15·10
Volume after shaking with water	15·15
Volume left unabsorbed by acid solution but absorbed by ammoniacal solution	0·35

When tested with the methane-free mixture it was found to absorb the carbon monoxide completely:

	c.c.
Original volume	41·95
Volume after shaking with ammoniacal solution (1)	23·35
Volume after shaking with ammoniacal solution (2) (fresh)	23·15
Volume after shaking with water	23·05
Volume after addition of oxygen	33·8
Volume after explosion	14·2
Volume after shaking with potash	14·2
Volume of CO not absorbed	Nil

A similar experiment on a different methane-free mixture gave 0·03 c.c., *i.e.* 0·06 per cent. of apparent methane, a figure within the limits of accuracy of reading.

For routine analysis three successive absorbers filled with the above solution are used; if, on shaking, any further contraction occurs in the third, the first is refilled with fresh solution and used as "No. 3," Nos. "2" and "3" becoming "1" and "2" respectively in rotation. This procedure ensures that the solution in the third pipette has not previously absorbed any carbon monoxide—a condition necessary to prevent incomplete absorption later.

Rate of absorption and vapour pressure of ammonia.—As in the case of the acid solution, 1 minute is sufficient for the absorption to take place, as is shown below. The effect of keeping the gases in contact with the solution is sometimes an increase of volume due to the vapour pressure of ammonia; this may be considerably greater than that of the hydrochloric acid in the acid solution. For this reason the gas should always be washed, after absorption of carbon monoxide, in a pipette filled with water. The magnitude of this effect varies with the details of preparation of the solution; it may be as small as 0·05 c.c., or as great as 1 c.c. The following results are examples showing the rate of absorption and the magnitude of the vapour pressure effect.

	c.c.		c.c.	
Volume after shaking 15 seconds with solution			37·20	
Volume after shaking 1 minute with same solution			37·30	No further absorption of CO
Voume after standing 5 minutes with same solution	23·00			
Volume after standing ½ hour	23·15			
Volume after washing with water	23·05	0·1 c.c. of NH_3 No further absorption of CO	36·3	1 c.c. of NH_3

	c.c.	
Volume after removal of CO_2 and oxygen.	38·25	
Volume after shaking 30 seconds with CuCl solution (1).	19·95	Volume of CO absorbed in first 30 seconds = 18·45 c.c., *i.e.* 99 per cent. of the total absorption in 4 minutes by same solution.
Volume after shaking with water.	19·80	0·15 c.c. NH_3 gas evolved by solution.
Volume after shaking 30 seconds with CuCl solution (1).	19·85	Volume of CO absorbed in second 30 secs. = 0·12 c.c. Volume absorbed in 1st minute = 18·57 c.c.
Volume after shaking with water.	19·68	0·17 c.c. NH_3 gas evolved by solution.
Volume after shaking 3 mins. with CuCl solution (1).	19·82	0·03 c.c. removed (a figure within the limits of reading error), *i.e.* practically all the CO is absorbed in the first minute.
Volume after shaking with water.	19·65	0·17 c.c. NH_3 gas evolved by solution.
Volume after shaking 30 seconds with CuCl solution (2) (fresh).	19·60	A further 0·13 c.c. absorbed by fresh solution in 30 seconds.
Volume after shaking with water.	19·52	0·18 c.c. NH_3 gas evolved by solution.

CUPROUS OXIDE IN SULPHURIC ACID CONTAINING β-NAPHTHOL.—Lebeau and Bedel (*Compt. Rend.*, 1924, **179**, 108; ANALYST, 1924, **49**, 451) recommend a suspension of cuprous oxide in sulphuric acid containing β-naphthol. Although, as the discoverers point out, the reagent can be kept in contact with mercury, a slow reaction appears to take place, with the evolution of gas amounting to about 0·02 c.c. of gas per hour per 100 c.c. of solution. This gas is absorbed by potassium hydroxide and is probably hydrogen sulphide, since the used solution has a faint odour of this gas. This effect can be greatly diminished by not using mercury in the pipette and, instead, protecting the surface of the liquid in the upper part of the pipette with a layer of B.P. paraffin. With absorption pipettes, such as that of Sodeau, in which the absorbent is kept in the pipette, this procedure is to be advised. Even with this precaution, however, the gas should always be shaken subsequently with water or potash.

The results obtained here show that the solution gives quantitative absorption of carbon monoxide within the limits of measuring error. Its efficiency, moreover, (unlike that of cuprous chloride) is not impaired by the previous absorption of considerable quantities of carbon monoxide.

Rate of absorption.—The rate of absorption is lower than that of cuprous choride, and is also largely governed by the amount of shaking.*

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* That this slowness of absorption and necessity for shaking was not due to the covering of the surface of liquid in the lower part of the pipette with a film of B.P. paraffin, is shown by the fact that similar results were obtained with the use of a pipette (containing mercury) from which B.P. paraffin was excluded.

The properties of this solution as regards the absorption of CO are demonstrated in the following experiments:

		c.c.	
Volume of gas after removal of CO_2 and oxygen		42·89	
Volume after shaking with Cu_2O suspension which had previously absorbed 50 c.c. of CO	3 mins.	25·36	76 per cent. absorbed.
	,,	20·12	98·5 ,, ,,
		20·06	99·2 ,, ,,
		20·01	100 ,, ,,
		20·01	100 ,, ,,
Volume after shaking 5 minutes with fresh suspension.		20·01	No further CO removed.
Volume after shaking with water.		19·82	Gases produced by absorbent removed.
Volume after shaking with fresh ammoniacal cuprous chloride.		20·13	Increase due to V.P. of NH_3.
Volume after shaking with water.		19·82	0·32 c.c. of NH_3 removed.
i.e. Volume of CO not absorbed by solution which had previously absorbed 50 c.c.		Nil	

12 minutes' continuous shaking required to bring about complete absorption.

		c.c.
Volume of gas after removal of CO_2 and oxygen.		40·16
Volume after contact with Cu_2O suspension.	½ minute's shaking.	39·05
	20 minutes' shaking.	29·9
	25 minutes' without shaking.	27·55
	10 minutes' shaking.	23·80

Rate of absorption with shaking, about 4 times that without shaking.
Volume absorbed in first ½ minute = 1·1 c.c. (*cf.* 15·25 c.c. with cuprous chloride).
About 30 minutes' shaking required for complete absorption.

		c.c.
Volume after removal of CO_2 and oxygen.		37·88
Volume after contact with Cu_2O suspension.	10 minutes' shaking.	26·6
	1½ hours' standing, with occasional shaking.	20·0
	5 minutes' shaking.	18·22
	,, ,,	17·80
	,, ,,	17·80

Rate of absorption with shaking, about 3 times that without shaking.

EFFECT OF THE AMOUNT OF PREVIOUSLY ABSORBED CARBON MONOXIDE ON THE EFFICIENCY OF THE THREE ABSORBENTS DESCRIBED.—This effect has been studied quantitatively. In the case of cuprous chloride the average figures from three or four samples of each solution have been taken.

The following table shows the relation between total volume of carbon monoxide previously absorbed by a sample of each solution (column 1) and the volume of carbon monoxide that that sample fails to take up.*

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* In all cases this residual carbon monoxide was mixed with 15 to 25 c.c. of hydrogen and nitrogen.

Volume of CO previously absorbed by 100 c.c.	Volume of CO left behind (in presence of 20 c.c. neutral gas).		
	by acid cuprous chloride.	by ammoniacal cuprous chloride.	by Cu_2O suspension.
c.c.	c.c.	c.c.	c.c.
0	0·3	0·0	0·0
1	0·3	0·0	0·0
3	0·3	0·0	0·0
5	0·4	0·0	0·0
10	0·4	0·0	0·0
15	0·4	0·1	0·0
20	0·5	0·1	0·0
30	0·5	0·1	0·0
40	0·5	0·1	0·0
50	0·6	0·2	0·0
60	0·7	0·2	0·0
80	0·9	0·2	0·0
100	1·0	0·3	0·0
150	1·3	0·4	0·0
200	1·8	0·6	0·0
500	—	—	0·2 0·0

Taking into consideration the experiments on rate of absorption, the poor absorption by old solutions would appear to be an equilibrium effect rather than one due to low rate of absorption. Except in the case of fresh ammoniacal solution, which does not contain more than 0·05 c.c. of dissolved CO per c.c., cuprous chloride does not absorb to completeness.

On the other hand, absorption by the cuprous oxide suspension has shown itself to be complete.

Summary.—(1) The acid solution of cuprous chloride does not, under any circumstances, effect complete absorption of carbon monoxide.

(2) The ammoniacal solution of cuprous chloride, if it has not previously absorbed more than about 5 per cent. of its own volume of carbon monoxide, does effect complete absorption.

(3) Previously absorbed carbon monoxide interferes with the completeness of the absorption in the case of both these solutions.

(4) Cuprous oxide in sulphuric acid containing β-naphthol gives complete absorption of carbon monoxide, even when it has previously absorbed considerable quantities. Its action, however, is slower than those of the chloride solutions.

PART II.

Advantages of Using Hot Reagent.

By T. CARLTON SUTTON, M.Sc., F.Inst.P., and H. R. AMBLER, B.Sc., A.I.C.

Cuprous oxide suspended in a mixture of β-naphthol and sulphuric acid* is found to absorb carbon monoxide completely. When used in the cold, however, more than ten minutes', and sometimes as much as half an hour's shaking, is needed to secure this result.

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* Lebeau and Bedel (*loc. cit.*).

We find, however, that this defect may be overcome by using the reagent at a temperature of about 60° C. Absorption them takes place within 3 minutes.

The Sodeau apparatus used in the previous work was employed for the analysis. Three and a half metres of 30 S.W.G. Eureka Wire were wound round the lower bulb of the gas pipette and connected with a 20 volt main. This was sufficient to raise the temperature of the bulb and absorbent to about 60° C. and maintain it at that temperature.

After carbon dioxide and free oxygen had been removed, the gas sample was passed into the pipette in the customary way, was duly shaken and, on removal into the jacketed "reading burette," was allowed to stand so as to regain the initial temperature. Thirty seconds were found to be sufficient for this purpose. Some typical absorptions showing the effect of warming and of shaking are given in the following table:

		Burette readings	Per centage of CO absorbed.	Time of shaking required for complete absorption.
		c.c.	c.c.	
Room Temp. 15° C. to 20° C.	Vol. gas before shaking with absorbent	42·89	0	12 minutes.
	„ „ after 3 minutes' shaking with absorbent	25·36	76	
	„ „ „ 6 „ „ „ „	20·12	98·5	
	„ „ „ 9 „ „ „ „	20·06	99·2	
	„ „ „ 12 „ „ „ „	20·01	100	
	„ „ „ 15 „ „ „ „	20·01	100	
	Vol. gas before shaking with absorbent	40·16	0	About 30 minutes (contact without shaking not effective).
	„ „ after ½ minute's shaking with absorbent	39·05	7	
	„ „ „ 20 „ „ „ „	29·90	63	
	„ „ „ further 25 minutes' contact without shaking	27·55	77	
	„ „ „ „ 10 „ shaking	23·80	100	
	„ „ „ „ 10 „ „	23·80	100	
	Vol. gas before shaking with absorbent	37·88	0	At least 15 minutes (contact without shaking not effective).
	„ „ after 10 minutes' shaking	26·00	59	
	„ „ „ additional 1½ hours' standing with occasional shaking	20·00	89	
	„ „ „ further 5 minutes' shaking	18·22	98	
	„ „ „ „ 5 „ „	17·80	100	
	„ „ „ „ 5 „ „	17·80	100	
Temp. 60° C. to 70° C.	Vol. gas before shaking with absorbent	38·65	0	3 minutes.
	„ „ after 1 minute's shaking	34·10	31	
	„ „ „ 3 „ „	24·20	99·9	
	„ „ „ 5 „ „	24·18	100	
	„ „ „ 7 „ „	24·18	100	
	Vol. gas before shaking with absorbent	29·30	0	1½ minutes.
	„ „ after ½ minute's shaking	23·10	48	
	„ „ „ 1½ „ „	16·54	100	
	„ „ „ 2½ „ „	16·54	100	

We have been using this method for some of our routine work and would recommend it for careful consideration to anyone who deals habitually (as we do) with mixures containing, say, carbon dioxide, carbon monoxide, oxygen, nitrogen, hydrogen, and methane.

Our thanks are due to the Director of Artillery for permission to publish this paper.

RESEARCH DEPARTMENT,
ROYAL ARSENAL, WOOLWICH.

The Examination of Charred Documents.

BY C. AINSWORTH MITCHELL, M.A., F.I.C.

(*Read at the Meeting, February* 4, 1925.)

THE special difficulties in an examination of documents become incomparably greater when the paper or other groundwork material has been injured by fire, and then it is often only by the most careful manipulation that it is possible to decipher any characters that may be present, or to ascertain the medium in which they were made.

The first scientific investigation of the subject appears to be that made by Sir Humphry Davy (*Phil. Trans. Roy. Soc.*, 1821, 191), when in 1819 he made a series of experiments to discover the best means of dealing with the charred Greek and Roman papyri which had been recovered from the ruins of Herculaneum where for over seventeen hundred years they had lain buried beneath layers of volcanic ash and tufa from Vesuvius. This had excluded the air, and the separate leaves of papyrus had become matted into compact scorched masses into some of which water had permeated, turning them black. Of the various methods of separation tried, that found to be most successful was to saturate the caked masses with chlorine or iodine vapour and then to heat them very gradually so as to expel the gas from between the individual leaves. Finally these were coated on the back with a solution of glue gelatinised by the addition of alcohol. In this way, fragments of a large number of papyri were separated and rendered legible, and it was proved by means of tests with potassium ferrocyanide that the inks upon them were not iron-gall inks.

It is, of course, now well known that all the inks upon early papyri were carbon inks (*cf.* Lucas, ANALYST, 1922, **47**, 22), but, had the inks on these Herculaneum MSS. been of the more modern type, they could hardly have escaped being bleached by the action of the chlorine. Davy mentions that he tried carbon dioxide as a separating agent, but did not obtain such good results with it as with chlorine.

Restoration of Iron Inks.—The method of testing for iron in inks by means of ferrocyanide was first used by Blagden in 1787 (*Trans. Roy. Soc.*, 1787, **77**, 451) in his experiments to ascertain the nature of the inks on ancient parchments from the ninth century A.D. onwards. On the whole, it gives more satisfactory results than treating the parchment or paper with a decoction of galls, which was described by Canneparius, in 1660, in his book on inks (*De Atramentis*), but a solution of gallic acid is preferable to gall nut extract for restoring writing on parchment MSS. rendered illegible by age or by scorching, and was recently found effective for the purpose by Matthews (Analyst, 1924, **49**, 516). In the case of charred or incinerated paper, however, neither ferrocyanide nor gallic acid is satisfactory, since the coherent fabric of char or ash is too porous, and the reagent spreads through the mass, making it so pulpy that it is difficult to handle.

I therefore made experiments with various reagents in the form of vapour. Nitric oxide will sometimes bring out the writing on calcined fragments, evidently through its combination with the ferrous sulphate reduced from the ferric basic sulphate in the burning process. This method is very uncertain, however, and it is much better to use thiocyanic acid vapour (obtained by heating a solution of a thiocyanate with hydrochloric acid). Under favourable conditions the reaction is very effective, a dark iron thiocyanate being formed from the residual iron from a writing. For example, quite illegible writing upon dark brown scorched paper was thus developed in very dark characters.

The reaction is an extremely sensitive one, and in a series of test experiments I have found it capable of detecting as little as 1 part of iron in 300,000. It is not surprising, therefore, that it does not give good results when the paper itself contained much iron.

Methods of Calcining.—It is only in exceptional cases that charred documents can be deciphered without continuing the calcination one or more stages further, and the difficulty I had to overcome was to be able to stop the process at any desired stage of charring, for some characters show up best on a light brown char, others on grey or white ash. The method I ultimately adopted was to place the fragment between two pieces of fine wire gauze and to hold these by means of the crucible tongs in a Bunsen flame. From time to time this gauze holder is withdrawn from the flame and the fragment examined, and in this way not only does the calcination proceed evenly, but the curling up of the edges of the paper is prevented to a large extent.

Differentiation of Pigments by Calcination.—Apart from its use in the development of illegible writing in ink or pencil, or of printed or typewritten characters, this method of calcining can also be used as a means of differentiating different pigments on unburnt paper.

Ordinary writing inks, on ignition, leave a residue probably consisting of a mixture of ferrous and ferric salts, and these show a different appearance on the charred ink according to the amount of iron in the ink. The intensity of the thiocyanic acid reaction may then afford a further means of differentiation.

Pigments.—Graphite is very difficult to burn away, even in the form of the thin deposit from a pencil. With moderate ignition a pencil mark is readily developed upon charred paper, and the microscopical appearance of the black line of residual graphite may be very characteristic. For instance, Rowney's indelible black pencil leaves a pale dark grey line on a grey ash from the paper, quite different from that given by the old Borrowdale pencils.

The silver striations due to the silicious impurities in the graphite or in the clay of the pencil, can usually still be clearly discerned in the calcined lines, and enable one to ascertain which of two intersecting lines is uppermost (*cf.* ANALYST, 1922, **47**, 386).

Copying Ink Pencils.—I have already shown (ANALYST, 1917, **42**, 3) that the pigments of these violet pencils can be differentiated on paper by various reactions based on the difference in their composition, and I have given a table showing that their pigments can be grouped into different classes in accordance with their relative contents of graphite, kaolin clay and violet dye.

Those pigments containing a considerable amount of graphite yield a well-defined line of graphite after calcination, whereas those free from graphite may show a white streak (due to alumina) or a grey ash, or, if the calcining is done too rapidly, leave no visible residue. The iron present also influences the result.

The following results are typical of those obtained:

Copying Ink Pencil.	*Result obtained.*
Eagle Atlas 824	Grey characters on white ash.
"Atlas" 823	Ditto.
Faber's	Brick-red characters on grey ash.
Duplex (American Pencil Co.)	Fawn-brown characters.
Hardtmuth's	Greyish graphite streak on white ash.
Hessin's	Black graphite streak on white ash.
Japanese	Dark graphite streak.
Kurz	Black graphite line on white ash.
Swan	White on grey ash, or nothing.

The last result is interesting, for it explains why in the case of *Rex* v. *Wood* (in which charred fragments of paper were found in the grate of a room where a woman had been murdered) so little of the writing remained legible. I have tested writing done with the "Swan" copying-ink pencil found on the prisoner, and it behaved in exactly the same way. The analysis of the pigment explained the reason of this difference; alumina but no graphite is present.

Another interesting point about the calcination of writing in copying-ink pencil is that if the pigment contains graphite the test enables one to decide which of two intersecting lines is on top. This is quite impossible with any degree of certainty when examining the pigment when freshly written (see Fig. 1), but on burning away the violet dye the black lead remains and the continuity of the silver striations in the lines becomes apparent, just as in the case of writing in ordinary black-lead pencil (see Fig. 2). These photo-micrographs were made by Mr. T. J. Ward under a magnification af 20 diameters.

Coloured Pencils.—The calcination test will frequently distinguish between the pigments of different pencils in writing on paper. In some cases the iron in the pigment is the characteristic ingredient; in others, alumina and occasionally black lead. The following results are typical of those obtained:

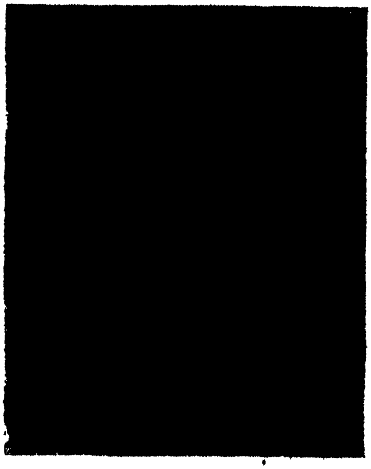

Fig. 1.
Marks with Copying Ink Pencil,
× 20.

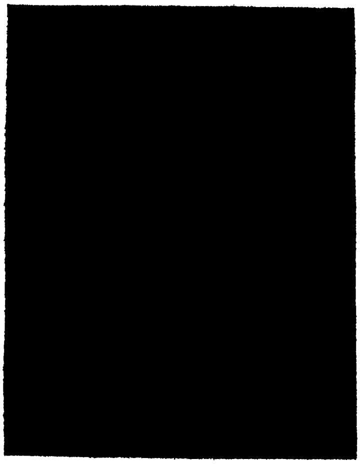

Fig. 2.
Marks with Copying Ink Pencil,
after calcination of paper, × 20.

Blue Pigments.	*Result of Calcination.*
American Pencil Co. solid pencil	Dark characters as incrustation completely obliterated to a white ash.
American Duplex copying ..	Faint grey on white ash; barely legible.
Faber's blue copying ..	Faint red on grey ash. Intensified to chocolate brown by HCNS.
Swan Pencil Co.	White on grey ash.
Red Pigments.	
Cohen's red pencil ..	White on brown charred paper.
Faber's red pencil ..	White on brown charred paper.
Chamber's red pencil ..	Dark metallic characters on grey ash.
Red solid pencil	Writing completely obliterated.

The foregoing results are sufficient to prove that the method could be used as a means of identifying these pigments in a given piece of writing, and need only involve the destruction of a word or two of the document.

TYPEWRITING.—The earlier typewriting inks, which consisted of an aniline dye mixed with a slowly drying medium, usually containing glycerin, are still in use. As might be expected, they leave no trace of the writing when the paper is calcined, and this distinguishes them from the more modern inks which are more of the nature of printing inks, and contain lampblack, colour lakes, and oil-soluble dyes. Those containing gasblack or other forms of lampblack can be quite clearly developed by cautious ignition between the wire gauze support, but if the heating is continued too long the carbon soon burns away. Typewriting inks containing coloured lakes may also leave a characteristic residue.

PRINTING INKS.—Black printing inks, which contain lampblack or gasblack, behave like the analogous typing inks. This form of amorphous carbon is not completely burned away in a short time until the temperature reaches 305°–310°, and as the carbonised paper is burned away long before that temperature is reached, it is quite a simple matter to develop printed characters on a white ash if the calcination is carefully followed.

Blue inks containing Prussian blue may leave a red residue due to the iron present, and this can be intensified by means of thiocyanic acid vapour.

Red printing inks, which frequently contain an organic pigment in the form of a lake, may also give very characteristic results. The organic pigment, of course, burns away, but the alumina may be left as a brilliant white deposit on the black char of the paper. Other red printing inks, containing an iron pigment, leave a brown deposit on a grey ash, and pure vermilion ink leaves black characters on a dark grey ash.

Illustrative Case.—The difficulty of the possibility of deciphering printed matter on a charred paper was of primary importance in a case in which I was consulted three years ago. A cashier was charged with appropriating a large sum of money in bank notes that had been entrusted to him. His defence was that in clearing away a pile of waste papers he had accidentally burned the pile of notes, but his story was not accepted, and he was sentenced to a term of imprisonment. He appealed, and asked that the ashes of his bonfire, which he had kept, might be searched. When I examined these ashes I found that less than 20 per cent. consisted of large charred fragments exceeding a half square inch in area, and less than 25 per cent. of still smaller fragments, the remainder being composed of dust and office débris. I estimated the number of large fragments at over 30,000.

It was necessary to examine these individually by oblique light, and, when there was the slightest indication of pattern, the selected fragments were examined more closely, frequently by calcination. By this means about ten fragments showing patterns which could be recognised as forming portions of bank notes could be identified, and ultimately I discovered one fairly large piece upon which the head in the medallion of a note could be unmistakably recognised.

Most of the fragments in the heap on which printed characters could be deciphered were on highly clayed paper. The larger fragments contained nearly 35 per cent. of mineral matter, and the smaller about 28 per cent. The paper upon which Bank of England notes are printed yielded a non-coherent residue containing 8·08 per cent. of mineral matter. These differences were quite sufficient to account for the difficulty of discovering only a few fragments of bank notes, although a bundle had been burned. The only reason why any portions at all of them were found was that they had been calcined in a compact mass, so that the inner portions would be more protected from the flame and thus leave a more coherent ash. This was proved by experiments with the bank note paper. This evidence in conjunction with the production of fragments of actual charred notes convinced the Appeal Court, and the sentence on the accused was quashed.

STRENGTHENING AGENTS.—The case described also shows the difficulty that may be experienced in calcining paper poor in mineral matter. The charred remains of ordinary magazine paper and the like, being heavily loaded with clay, can be developed at once, for it forms an excellent background for printed or written characters. But when only a small amount of mineral matter is present, it is essential to supply it by treating the partly charred fragments with a solution of a suitable salt, and then drying it before calcination. A 10 per cent. solution of aluminium acetate gives very good results; so does a solution of thorium acetate of the same strength. Habermann (*Zeitsch. anal. Chem.*, 1909, **48**, 729) expressed the view that thorium nitrate was unsuitable for this purpose, owing to there being some risk of its oxidising the graphite of lead pencil marks. In view of the difficulty of burning graphite away, I applied tests to ascertain whether such oxidation actually would happen, and I have to thank the Welsbach Light Company for kindly sending me specimens of pure thorium nitrate solution (20 per cent.) and of pure cerium nitrate as used in the preparation of incandescent gas mantles. I found that charred fragments of paper poor in mineral matter leave a compact coherent ash after treatment with thorium nitrate, and that graphite pencil marks are not removed during the calcination. Cerium nitrate, on the other hand, is of little use for the purpose, for it disintegrates the ash fabric into small irregular fragments.

The simplest method of preserving the calcined fragments is to attach the underside to a microscope slide by means of a thin film of seccotine or, if both sides are wanted for examination, the fragments may be sprayed on each side with a transparent spirit varnish and preserved between two pieces of tracing paper.

PHOTOGRAPHIC METHODS.—It is frequently possible to decipher dark charred fragments by direct photography with oblique lighting, which often reveals more than can be seen by the eye. According to Dennstedt and Voigtländer (*Lehrbuch der gerichtlichen Chemie*, 1906, p. 120) light grey characters on black backgrounds of char may be brought out well by the use of a red filter and colour-sensitive plate with oblique lighting.

About two years ago an official method of deciphering charred documents by means of photography was published in the United States (Davis, *U.S. Bureau of Standards Sci. Papers*, 1922, **18**, (454), 445; *Abstr. Analyst*, 1923, **48**, 241). This is based upon the principle that a sensitised plate, when left in close contact with a charred fragment of paper, is acted upon by some of the products of the combustion, but that those parts of the plate in contact with any latent writing or printing will be protected from this action. Although the method is a very slow one, at least a week, and often a month, being required to obtain a record of any characters present, it might be useful under certain conditions when calcination is not permissible. Unfortunately my experiments, and those of Mr. T. J. Ward, have shown that one cannot be certain of getting a successful result however long the exposure is continued. Very much depends upon the degree of charring to which the paper has been subjected. For example, in two typical instances pieces of the same paper with characters in the same printing ink were charred for different periods, then left in close contact with Ilford Rapid Plates for three weeks. The one more lightly charred gave a faint indication of the printed characters, whereas the second piece showed an outline of the paper, but no suggestion of the letters.

Many years ago Dr. Russell showed that ordinary printed matter might record itself on a photographic plate, probably on account of certain peroxides formed by the driers in the printing ink. But the results are just as capricious as with charred fragments. For example, Mr. T. J. Ward obtained an excellent contact reproduction of a fragment of a newspaper within a week, whereas I left portions of another periodical in contact with an Ilford Rapid Plate for a period of five weeks without obtaining any indication of characters on the plate.

My experiments with the charred printed matter were made immediately after the charring, but, as the author of this photographic method points out, its success largely depends upon the contact with the sensitive plate being made as soon as possible. The poor results that I obtained under the most favourable conditions, for I made ten exposures for varying periods of time and only twice obtained a poor record of the printed characters, show that the method, though interesting, is obviously too limited in its application to be of much practical use.

I wish to thank Mr. W. B. Murdoch for specimens of printing in different kinds of ink, and Mr. T. J. Ward for his assistance in the photographic work.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

THE FAT OF GOATS' BUTTER.

REFERRING to Mr. Richmond's note (ANALYST, 1925, 62), we would repeat that the argument in our paper (ANALYST, 1924, 49, 509) was based on the fact that we knew of cases where goat's butter had been sold as "butter."

Whether or not goats' butter is legally butter is a point which is somewhat obscure, although we ourselves incline to the view that, as the law stands at present, it may be described as "butter."

It was with the object of directing attention to the present somewhat unsatisfactory legal position of this product that our notes were mainly written, as a perusal of our conclusions will show.

Mr. Richmond's next point is, "that by the use of accepted methods and especially by the use of formulæ," of which he is the author, we have formed the conclusion that goats' butter might be condemned as adulterated with coconut oil. (We presume that he has understood our meaning, *viz.* if the sample were received by an analyst as "butter.")

Mr. Richmond is quite correct in assuming that we used one of his formulæ in the manner he indicates. We were astonished, however, to find that a different meaning existed from the one we attributed to the words defining P′ in the formula which he gives on p. 249 of *Dairy Chemistry*, 3rd Edition. Doubtless Mr. Richmond was equally surprised. A correct quotation of the formula and directions for its use, both as regards wording and punctuation, taken from that page of his book is:

"Should the Polenske figure exceed the maximum given in the table the quantity of coconut oil present may be deduced from the formula

$$C = \frac{P - P'}{14{\cdot}4} \times 100.$$

C = percentage of coconut oil.
P = Polenske figure.
P′ = mean Polenske figure from the table for a figure equal to the Reichert-Wollny figure found + half the Polenske figure."

Expressed algebraically, we consider that the obvious meaning of Mr. Richmond's words is that $P' = \phi(a) + b$, whereas actually they are intended to mean $P' = \phi(a + b)$, where the symbol ϕ represents "function."

We fail to see how the unintentional omission of the words "for a figure" affects the meaning of the directions in any way, and in any case our interpretation of the meaning of the words was made before the misquotation complained of.

Incidentally, in his remarks Mr. Richmond also misquotes his book, as the word we have put in italics does not appear therein, *viz.* "*calculated* for a figure."

However, we frankly admit that we did not use this formula in the manner in which Mr. Richmond now makes clear it should be applied, and we thank him for calling our attention to the mistake.

Naturally the correct use of the formula calls for a modification of the view we had expressed, *viz.* that samples of goats' butter received by an analyst as "butter" might be regarded as cows' butter and coconut oil, as the calculated Reichert-Wollny figures for the apparent original butter would be too high in a great many cases for this to be possible.

Further, it still demonstrates what we have already stated—that the formula cannot be made to apply to goats' butter. We are well aware that it was not designed to do so, but we have endeavoured to place ourselves in the position of an analyst who received such samples as "butter." In the case of certain mixtures of goats' and cows' butter, which might be described as "butter," we still maintain that, although other determinations might lead to a contrary view, the inference from the Reichert-Wollny, Polenske and Kirschner figures is that coconut oil had been added, and therefore so far as such "butters" are concerned, these figures are unreliable. This can be seen from the figures these determinations would give if applied to "butters" made by blending the following percentages of our average goats' butter with average cows' butter. This has been verified by experiment.

The average of our results for goats' butter would give:—R.W., 26·44; P., 7·3; K., 18.

The average for cows' butter is taken as:—R.W., 28·4; P., 2·3; K., 24.

From mixtures of these, approximately the following figures would be obtained on analysis:

	Percentage of Goats' Butter.			
	20	30	40	50
R.W. No.	28·01	27·81	27·62	27·42
P. No.	3·30	3·80	4·3	4·8
K. No.	22·8	22·2	21·6	21

Application of the coconut oil formula to these figures would indicate the following:

Percentage of Coconut Oil indicated.			
3·82	7·15	10·63	14·1

Calculating the Reichert-Wollny figure of the apparent original butter from these figures would not arouse suspicion, but, of course, with higher percentages of goats' butter high calculated Reichert-Wollny figures would be obtained. For the above mixtures the figures are:

R.W. of apparent coconut oil	0·3	0·57	0·85	1·13
R.W. apparently due to butter portion ..	27·70	27·24	26·77	26·29
Calculated R.W. of original butter ..	28·80	29·34	29·95	30·61

With goats' butter of as low Reichert-Wollny value as those recorded by Trimen, the apparent percentage of coconut oil could, of course, be higher.

It appears to us that a person prosecuted for selling a coconut oil butter mixture might offer the defence that it was cows' and goats' butter, and, as such, was entitled to be called "butter."

In conclusion, may we say that our experience leads us to believe that goats' butter is not such a "rare commercial article" as Mr. Richmond states in his letter.

Frank Knowles.
John C. Urquhart.

BELLIER'S MODIFIED TEST FOR ARACHIS OIL.

I should like to direct the attention of other analysts to the fact that imported olive oil is now frequently condemned as containing 5 per cent. of arachis oil, owing to its having given a positive result in a rapid modification of Bellier's test. This modification consists in adding to the saponified oil 1·5 c.c. of Adler's acetic acid solution and omitting the hydrochloric acid from the alcohol. Under these conditions the test sometimes gives a cloudiness at or about 16° C. By the use of Luer's modification, however, in which 3 drops of glacial acetic acid are added to the saponified product (Analyst, 1913, **38**, 58) no turbidity results. In my experience a cloudiness or precipitate at 17° C. is indicative of the presence of arachis oil, but it is unsafe to base judgment on a turbidity at 16° C.

As this supposed detection of arachis oil is causing great loss to the sellers of olive oil, I should be glad to know the experience of other analysts as to the degree of accuracy of this modification of the test. F. F. Shelley.

Apothecaries' Hall, E.C.4.

PREPARATION OF NESSLER'S SOLUTION.

The note under the above title by Mr. H. Droop Richmond in the February issue reiterates a statement that is commonly encountered in the literature of water analysis, namely, that "The sensitiveness of Nessler's solution . . . usually increases with age." My own experience is quite at variance with this statement, and since reading it I have made a test, the method and results of which are sufficiently set out in the table below, to determine whether ageing had the effect alleged.

	2 c.c. of the solution added to 50 c.c. of water:		
Nessler's solution.	Containing 0·01 mgrm. NH_3	Containing 0·003 mgrm. NH_3	Ammonia-free and used to prepare foregoing standards.
1. 14 years old; stored in 2 c.c. sealed glass ampoules.	Distinct coloration, but less than No 2.	Coloration just discernible.	No coloration.
2. 6 months old; stored in stock bottle.	Distinct coloration, but less than No. 4.	Coloration quite definite.	No coloration.
3. 3 days old; from stock bottle.	Same as No. 2.	Same as No. 2.	No coloration.
4. 6 months old; from stock bottle and sensitised immediately before use.	Very distinct coloration.	Coloration marked and distinctly more than No. 2.	No coloration.

These observations were confirmed by an assistant who had no knowledge of the contents of the glasses. It is submitted that the tests show the statement to be inaccurate. The process of sensitising the Nessler solution, as is well known, consists in adding, to a suitable quantity withdrawn from the stock bottle, saturated mercuric chloride solution, drop by drop, with shaking, until a distinct yellow turbidity is produced, but it is not usually known that the effect remains at a maximum for only about 2 hours; this turbidity and coloration disappears when 2 c.c. of the solution are added to the 50 c.c. of water usually tested. It may be noted that the difference in colour intensity produced by a sensitive and an insensitive Nessler solution is much more marked when a quantity of ammonia of the order of 0·005 mgrm. is present than with larger amounts.

Robert C. Frederick.

Royal Naval Medical School,
R.N. College, Greenwich, S.E.10.

DYED ORANGES.

What appears to be a fresh example of ingenuity in colouring food to make it more attractive to the eye has recently been met with by the writer. Some oranges, which appeared to be ripe, were examined, and the redness of their skins was found to be due to artificial colour. A sample of the colouring matter said to be used for this purpose was proved to be a solution of chrysoidine, which, it may be mentioned incidentally, is also used for making bait appear more alluring to fish.

A point of distinction between dyed and natural oranges is that the calyx of the former is red, that of the latter green. On boiling the peel with water containing 10 per cent. of potassium bisulphite and a fragment of woollen fabric (nun's veiling) the fibres are coloured pale yellow in the case of natural oranges, and deep yellow with dyed oranges.

J. F. Liverseege.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

CITY OF BIRMINGHAM.

Report of the City Analyst for the Fourth Quarter, 1924.

During the fourth quarter of last year 1320 samples were submitted for analysis, of which 1118 were analysed under the Sale of Food and Drugs Acts. Of the food and drugs samples, 955 were bought informally (12 adulterated), and 163 were bought under the provisions of the Acts (9 adulterated).

Milk.—Of the 604 samples examined, 11 contained less than 11·5 per cent. of total solids. The percentage of adulteration was 1·8.

Butter.—One of 84 informal samples contained 19·5 per cent. of water. A subsequent formal sample contained only 13·9 per cent.

Margarine.—One of 106 informal samples was adulterated with boric acid (0·7 per cent.). Two formal samples bought as "Margarine mixed with Butter" contained less than 1 per cent. of butter. The vendor was cautioned.

Jam, Jelly.—Eight informal samples were free from boric or salicylic acid. One sample each of apricot, damson and raspberry jam contained glucose syrup, and two of them were labelled "made from refined sugar." The vendors were cautioned for the false labels.

Plant and Vegetable Manures.—Eight samples were examined for the Agricultural and Small Holdings Committee. One contained excess of insoluble phosphates (17·48 per cent. guaranteed, 23·3 per cent. found).

Mixed Poultry Corn.—One of 4 samples examined was composed of 33 per cent. of flint grit, 34 per cent. of maize, 32 per cent. of rye, and 1 per cent. of wheat. The vendor was cautioned, and a circular was sent by the Town Clerk to corn dealers in the city, pointing out that grit is not corn, and that "mixed corn" must consist of corn.

J. F. Liverseege.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

EXCESS OF QUININE IN PRESCRIBED MEDICINE.

On February 6 the proprietors of a drug store were summoned before the Islington Justices on the charge of supplying a medicine not in accordance with the purchaser's demand.

A prescription had been handed to the defendant requiring, among other ingredients, 2 grains of quinine sulphate in each half-ounce. The medicine was supplied and, on analysis, was found to contain an excess of 25 per cent. of quinine sulphate.

For the defence it was pointed out that there could be no intent to defraud in this case. The directors of the stores had made inquiries and had found that the quinine sulphate used had lost almost all its water of crystallisation. The result was that the medicine was found to contain more quinine sulphate (in terms of the B.P. article) than the amount of drug that was, in fact, put in. This was not put forward as an answer in law to the charge, but as grounds for mitigation.

A fine of £5 with £2 2s. costs, was imposed.

TURPENTINE SUBSTITUTE.

On February 27th a tradesman was summoned under the Food and Drugs Acts by the Shoreditch Borough Council, for having sold as "turpentine" an article that consisted entirely of petroleum derivatives.

The defendant pleaded ignorance of the matter.

Evidence was given by the inspector that on his instruction a boy was told to buy two pennyworth of turpentine. The boy had previously bought some for his mother. The mother of the boy stated that the "turpentine" previously bought had blistered her skin.

The defendant stated that he did not know what effect the contents of the bottle would have on the skin. He only knew that many children asked for "turps" without any definition, and others for "best turps."

Mr. Ray said that the defendant kept an oil shop, and that it was a general custom for the public to buy household remedies from such shops. The prosecution contended that if these shops sold things that were for use medicinally, they should not be allowed to claim that they were not subject to the Food and Drugs Acts. Turpentine undoubtedly came within the definition of a drug.

The Magistrate (Mr. Clarke Hall) pointed out that the defendant had admitted that it was his practice to sell this stuff unless the best turpentine was asked for. It was entirely a false description. Poor people came to him, and he sold them a substance that did them injury. Defendant must pay £20, which was the maximum penalty that the law allowed.

CIDER VINEGAR FROM DRIED APPLES.

U.S.A. Supreme Court Decision.*

In 1920 it was decided in an Ohio Court that 95 barrels of alleged apple cider vinegar sent by a New York company into the State of Ohio were adulterated and misbranded, for the reason that vinegar made from evaporated or dried apple products had been mixed and packed with and substituted wholly or in part for apple cider vinegar.

In 1922 the shippers appealed to the Circuit Court of Appeal on writ of error, and the judgment of the lower Court was reversed. The U.S.A. Government then appealed to the U.S. Supreme Court.

The facts not in dispute were that the claimants made apple cider and apple cider vinegar from fresh apples during the apple season from about September 25 to December 15, and during the remainder of the year they made these products from evaporated apples. In the evaporation process sulphur fumes were applied to prevent rot and discoloration, and about 80 per cent. of the water was removed by evaporation. In using the product for the preparation of cider vinegar, an amount of pure water substantially equivalent to that removed was added, and the sulphur was removed by the addition of barium carbonate. The traces of barium left in the extract did not constitute adulteration within the meaning of either of the paragraphs of Sec. 7 of the U.S. Food and Drugs Act, and were not alleged by the prosecution to be injurious or deleterious.

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* *U.S.A. Dept. Agriculture, Service and Regulatory Announcements*, No. 12367, pp. 194–205.

After being submitted to alcoholic and acetic fermentations the aqueous extract of the dried apples gave chemical results similar to those obtained from an analysis of cider vinegar from fresh apples, except that it contained the above-mentioned traces of barium.

After reviewing these facts the Supreme Court, in its judgment, cites the provisions of Sec. 6 of the U.S.A. Food and Drugs Act on labelling, and proceeds as follows:

"The statute is plain and direct. Its comprehensive terms condemn every statement, design, and device which may mislead or deceive. Deception may result from the use of statements not technically false or which may be literally true. The aim of the statute is to prevent that resulting from indirection and ambiguity, as well as from statements which are false. It is not difficult to choose statements, designs, and devices which will not deceive. Those which are ambiguous and liable to mislead should be read favourably to the accomplishment of the purpose of the Act. The statute applies to food, and the ingredients and substances contained therein. It was enacted to enable purchasers to buy food for what it really is. (*United States* v. *Schider*, 246 U.S. 519, 522; *United States* v. *Lexington Mill Co.*, 232 U.S. 399, 409; *United States* v. *Antikamnia Co.*, 231 U.S. 654, 665.)

"The vinegar made from dried apples was not the same as that which would have been produced from the apples without dehydration. The dehydration took from them about 80 per cent. of their water content—an amount in excess of two-thirds of the total of their constituent elements. The substance removed was a part of their juice from which cider and vinegar would have been made if the apples had been used in their natural state. That element was not replaced. The substance extracted from dried apples is different from the pressed-out juice of apples. Samples of cider fermented and unfermented made from fresh and evaporated apples, and vinegar made from both kinds of cider were submitted to and examined by the District Judge who tried the case. He found that there were slight differences in appearance and taste, but that all had the appearance and taste of cider and vinegar. While the vinegar in question made from dried apples was like, or similar to that which would have been produced by the use of fresh apples, it was not the identical product. The added water, constituting an element amounting to more than one-half of the total of all ingredients of the vinegar, never was a constituent element or part of the apples. The use of dried apples necessarily results in a different product.

"If an article is not the identical thing that the brand indicates it to be, it is misbranded. The vinegar in question was not the identical thing that the statement, 'Excelsior Brand Apple Cider Vinegar made from selected apples,' indicated it to be. These words are to be considered in view of the admitted facts and others of which the court may take judicial notice. The words 'Excelsior Brand,' calculated to give the impression of superiority, may be put to one side as not liable to mislead. But the words, 'apple cider vinegar made from selected apples,' are misleading. Apple cider vinegar is made from apple cider. Cider is the expressed juice of apples, and is so popularly and generally known. It was stipulated that the juice of unevaporated apples when subjected to alcoholic and subsequent fermentation is entitled to the name 'apple cider vinegar.' The vinegar in question was not the same as if made from apples without dehydration. The name 'apple cider vinegar' included in the brand did not represent the article to be what it really was; and, in effect, did represent it to be what it was not—vinegar made from fresh or unevaporated apples. The words 'made from selected apples,' indicate that the apples used were chosen with special regard to their fitness for the purpose of making apple cider vinegar. They give no hint that the vinegar was made from dried apples, or that the larger part of the moisture content of the apples was eliminated and water substituted therefor. As used on the label, they aid the misrepresentation made by the words 'apple cider vinegar.'

"The misrepresentation was in respect of the vinegar itself, and did not relate to the method of production merely. When considered independently of the product, the method of manufacture is not material. The act requires no disclosure concerning it. And it makes no difference whether vinegar made from dried apples is or is not inferior to apple cider vinegar.

"The label was misleading as to the vinegar, its substances, and ingredients. The facts admitted sustain the charge of misbranding.

"Judgment reversed."

Department of Scientific and Industrial Research.

FOOD INVESTIGATION BOARD.

Special Report No. 21.

THE "GAS" CONTENT AND VENTILATION OF REFRIGERATED HOLDS CARRYING APPLES.*

In a ship's hold carrying fruit the atmospheric conditions are influenced by the respiration of the fruit and by leakages from the hold, either by mass movement or by diffusion. The results of daily measurements of the carbon dioxide and oxygen concentrations in two "unventilated" holds during a voyage from Australia showed that the carbon dioxide concentration rose rapidly until more or less steady values were obtained, and remained steady until another rise occurred towards the end of the voyage, the oxygen concentration showing inverse changes. Assuming the leakage to be entirely by mass movement, a daily exchange of at least 10,000 cb.ft. per day between the atmospheres inside and outside the hold must have taken place. Effects of ventilation are only temporary. The observed value of the sum of the carbon dioxide and oxygen concentrations in the hold (21·0 for ordinary air) remained practically steady throughout the voyage in the two holds examined, and was between 21·3 and 21·6, due either to the respiratory quotient being greater than 1, and/or to leakage by diffusion of oxygen in at a greater rate than of carbon dioxide out. During the last few days of the voyage the (CO_2+O_2) value fell in one hold (data not available for the second). Carbon dioxide production had then increased, and must have been accompanied either by a still more rapid increase of oxygen consumption, or a decreased rate of leakage by diffusion (not by mass movement). D.G. H.

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* Obtainable from Adrastal House, Kingsway, W.C. Price 1s. 3d. net.

Special Report No. 22.

"BROWN HEART IN AUSTRALIAN APPLE SHIPMENTS."*

THE accumulated data point conclusively to the appearance of brown heart being due to the accumulation of carbon dioxide in the presence of a certain amount of oxygen in the ship's hold. In observations made in two ships' holds the safety limit of 10 per cent. of carbon dioxide was reached at the end of the first fortnight, and in holds showing high percentages of carbon dioxide (over 15 per cent.) the apples have invariably been found to be diseased. Moreover, in 10 common Australian varieties of apples brown heart has been induced by restricted ventilation at the temperature of ships' holds, the extent of disease increasing with the rising carbon dioxide percentage. Orchard conditions and factors inherent to the fruit, and its degree of maturity, may govern susceptibility to the disease. When apples are stored in holds, as noted in Report No. 21, the proportion of carbon dioxide produced rises steadily, reaches a level, and then, after a considerable steady interval, rises again. This phenomenon can be reproduced in sealed boxes of apples, and in the case of 150 lbs. of fruit thus stored, and maintained at an even temperature of 34–38° F., the percentage of carbon dioxide rose to 23–25 per cent., remained steady for 18 days, and rose again at the rate of 0·5 per cent. per day for 40 days until conclusion of the experiment. This would appear to be the result of the setting up of pathological conditions in the fruit, bringing about increased rate of carbon dioxide production. Experiments are being carried out to determine definitely whether the occurrence of brown heart is accompanied by increased rate of carbon dioxide production.

D. G. H.

* Obtainable from Adrastal House, Kingsway, W.C.2. Price 1s. 3d. net.

Dangerous Drugs Convention.*

THE International Drugs Convention, which will come into force when ratified, contains the following enactments:—

Medicinal opium means raw opium, powdered, granulated or mixed with indifferent materials, dried at 60° C., and containing less than 10 per cent. of morphine.

Morphine, cocaine, ecgonine and *diacetylmorphine* are defined as the substances represented by their chemical formulæ, and *Indian hemp* means the dried flowering or fruiting tops of the pistillatic plant, *Cannabis sativa* L., from which the resin has not been extracted, by whatever name they may be called in commerce.

Raw opium, coca leaves and other dangerous drugs, including all official and non-official preparations, such as anti-opium remedies, containing more than 0·2 per cent. of morphine and 0·1 per cent. of cocaine, or any alkaloid of opium or coca leaf, are only to be produced, distributed, imported and exported for medicinal and scientific purposes, and, to ensure this, the contracting parties are to exercise due control. Accurate records of manufacture by licensed persons are to be kept. Emergency supplies to the public, by pharmacists, of preparations containing a maximum of 25 cgrms. of opium are allowable at the discretion of the contracting nation.

D. G. H.

* *Pharm J.*, 1925, **114**, 226–227.

Italian Regulations on Colouring Matters.*

REVISED regulations for the use of colouring matters for non-textile purposes came into force in Italy on January 1, 1925, by a Royal Decree.

Colours containing arsenic, antimony and several other compounds may not be used in the preparation of food or for colouring paper in which food is to be packed, or for colouring containers for storing foods or beverages. These pigments may not be used for dentifrices or for colouring toys, but certain compounds of lead, antimony, barium, tin and zinc are allowable for toys. The use of colours containing arsenic, antimony, chromium, tin, zinc, uranium, and cyanogen derivatives is prohibited in soap, except in the case of certain medicated soaps. Arsenical pigments may not be used for colouring fabrics for upholstery, clothes, tapestry, artificial flowers, fruits or leaves, or for wall paper, candles, stationery, lamp-shades, etc.

Cosmetics, dyes and other preparations for colouring the skin must have the name of the manufacturer or seller on the package. If they contain poisonous substances, such as compounds of lead, silver or copper, or paraphenylenediamine, pyrogallol, aminophenol and the like, the exact nature of the contents must be printed on the label, and a yellow band must be attached on which is printed, in characters at least 4 mm. high, an intimation that the contents of the package may be injurious.

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* The Decree (in Italian) may be seen at the Department of Overseas Trade, 35, Old Queen Street, London, S W.1.

The Institute of Chemistry.

FORTY-SEVENTH ANNUAL GENERAL MEETING, 2ND MARCH, 1925.

THE President (Prof. G. G. Henderson), after referring to the loss sustained by British Chemistry by the death of Sir George Beilby, one of the Past Presidents, announced that the Council had invited the co-operation of the Society of Chemical Industry and the Institute of Metals, of which Sir George had also been President, in establishing some fitting memorial in his honour. Prof. Henderson also paid a high tribute to Sir James Dobbie, Government Chemist, to Mr. Otto Hehner, who had achieved a world-wide reputation for his investigations in the chemistry of food and drugs, and to Prof. G. D. Liveing, who had laid the foundations for the great school of chemistry, which had since been developed by Sir William Pope at Cambridge.

At the conclusion of the formal business, the President, supported by Prof. Thomson, made a presentation, on behalf of the Fellows and Associates, to Mr. Richard B. Pilcher on completing thirty years as Secretary and twenty-five years as Registrar and Secretary of the Institute. In the evening, the Council entertained Mr. and Mrs. Pilcher and their family to dinner at the Hotel Russell, followed by a reception.

The Officers and Council for the year ending March, 1926, were elected as follows:—

President.—George Gerald Henderson.

Vice-Presidents.—Edward Charles Cyril Baly, Edward Richards Bolton, Alfred Chaston Chapman, Thomas Slater Price, Arthur Smithells, and Edward William Voelcker.

Hon. Treasurer.—Patrick Henry Kirkaldy.

General Members of Council.—Leonard Archbutt, Francis William Frederick Arnaud, Hugh Charles Loudon Bloxam, Arthur Jenner Chapman, Frederick Daniel Chattaway, Harold Govett Colman, William Murdoch Cumming, Alfred Vincent Elsdon, Thomas Gray, Richard Henry Greaves, Arthur James Hale, Isidor Morris Heilbron, William Henry Lewis; Thomas Macara, Hamilton McCombie, Robert Selby Morrell, Robert Howson Pickard, Benjamin Dawson Porritt, Frank Lee Pyman, William Rintoul, William Henry Roberts, William David Rogers, Fred Scholefield, Harry Silvester, Arthur Slator, Jocelyn Field Thorpe, and James Fowler Tocher.

Censors.—Alfred Chaston Chapman, Sir Herbert Jackson, Gilbert Thomas Morgan, and Sir Robert Robertson.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Detection of Putrefying Flesh by means of Alcohol. Lüttge and Mertz. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 451–452.)—Amino acids are a constant decomposition product of flesh, and these finally yield ammonia and carbon dioxide. If, therefore, the amount of ammonia liberated on treatment with sodium hydroxide is high, it is indicative of putrefaction. The method is to extract with alcohol, which coagulates the albumin and dissolves the amino acids, which may be identified or determined. From 0·5 to 0·8 grm. is treated with 10 c.c. of alcohol and tested qualitatively for amino acids by the addition of a 1 per cent. alcoholic solution of ninhydrin or with α-naphthylisocyanide or, quantitatively, by the Sörensen titration or the micro-Kjeldahl method. Any considerable amount of amino nitrogen indicated by these tests shows decomposition. H. E. C.

Determination of Maltose in the Presence of Glucose. M. P. Nottin. (*Bull. de l'Assoc. des Chim.*, 1924–25; **44**, 143–145; *J. Inst. Brew.*, 1925, **31**, 145.)—The method depends upon the fact that maltose reduces Barfoed's solution to a small but definite extent. In the case of solutions of a concentration of 1 per cent. this reducing power of maltose was found to be 0·07 in terms of glucose as unity. Since the corresponding value with Fehling's solution is 0·572, the ratio of maltose to glucose in a solution containing not more than 0·5 per cent. of the reducing sugar (in terms of glucose) can be calculated from the results obtained with the two reagents the results of determinations by this method agreed with those found polarimetrically, and by fermentation with a yeast that does not ferment maltose.

The Dextrose and Fructose Ratio in Honey. A. Gronover and E. Wohnlich. (*Zeitsch. Unters. Nahr. Genussm.*, 924, **48**, 406–411.)—Investigation has been made of the methods and conclusions of Auerbach and Bodlander (ANALYST, 1924, **49**, 389), who, determining dextrose iodimetrically and fructose by difference, state that the ratio of dextrose to fructose in genuine honey is never less than 100:106. The method is generally accurate, and the results on known mixtures are satisfactory, the accuracy of the process being limited to about 1 per cent. of dextrose, but these authors do not confirm the limiting ratio given by Auerbach and Bodlander. With 33 honeys known to be genuine, the ratio was less than 106 in 16, and in 7 less than 100. None of these samples gave a positive result in Fiehe's reaction. H. E. C.

Non-volatile Acids of the Blackberry. E. K. Nelson. (*J. Amer. Chem. Soc.*, 1924, **47**, 568–572.)—Optically active iso-citric acid constitutes about five-sixths of the total acids of the blackberry, which contains also an appreciable proportion of *l*-malic acid and traces of oxalic, succinic, and citric acids. T. H. P.

Reducing Substances in Different Vinegars. G. Reif. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, **48**, 424–435.)—The purity of artificial acetic acid, whether prepared from acetylene or from wood distillation, can be judged by its reducing action on permanganate. This action is particularly due, in the case of acid prepared from acetylene, to traces of toxic compounds, such as crotonaldehyde and crotonic acid. If 1 c.c. of a 1 per cent. solution of permanganate be added to 5 c.c. of the acid diluted with 15 c.c. of water, crude wood acid (9·6 per cent.) decolorises the permanganate instantly; technically pure (50 per cent.) acid only after a few minutes; and chemically pure acid remains pink for 15 minutes. The action on permanganate of a large number of compounds when added in quantities ranging from 0·005 grm. to 0·2 grm. per 5 c.c. is tabulated, the most important reducing substances being crotonic acid and the cresols; butyric and oxybutyric acids, furfural and acetone, also reduce fairly rapidly. In wine vinegar the reducing substance is acetyl-methyl-carbinol, which substance also occurs in apple, raspberry and malt vinegars. Its detection, by Farnsteiner's method, serves for the differentiation of wine vinegar from spirit vinegars, or for the detection of wine vinegar in vinegar essences.

Note.—Farnsteiner (*Zeits. Unters. Nahr. Genussm.*, 1899, **2**, 198) distils 50 c.c. of vinegar, collecting 10 c.c. which is neutralised, mixed with 10 c.c. of Fehling's solution, and allowed to stand at the ordinary temperature. Spirit vinegars show no change of colour, but wine vinegars give a green coloration in about an hour, followed by deposition of copper oxide. H. E. C.

The Fatty Acid Constituents of some Natural Fats. I. The Oils from the Coconut. E. F. Armstrong, J. Allan and C. W. Moore. (*J. Soc. Chem. Ind.*, 1925, **44**, 63–68T.)—A series of fractions was obtained of which every alternate member is claimed to consist of a pure ester, and thus allowed of its identity being definitely established, so that there was no assumption in the identification of the middle fractions. The preliminary treatment and separation of the fatty acids before esterification depends on their iodine values, and esterification is carried out as follows:—Five hundred grms. of fatty acids are dissolved in 1500 grms. of absolute alcohol, and 150 grms. of concentrated sulphuric acid added (100 grms. in the case of liquid fatty acids). After being boiled for 3 hours the mixture is cooled, the supernatant layer of esters separated, the alcoholic liquor concentrated to about half its bulk, cooled, poured into water, and the esters extracted with petroleum spirit, those previously extracted being afterwards added. The solution is washed with water, mineral acid, and sodium hydroxide solution to remove free fatty acids (which should be recovered, esterified and united to the bulk). The solvent is then removed, and the ethyl esters distilled under reduced pressure, and subsequently fractionally distilled, still under reduced pressure. Reference should be made to the original paper for details of the fractions obtained and their examination. The composition of the coconut kernel oil (from an average sample of the meat of 200 African coconuts with the rind removed) and that of the "parings" oil, *i.e.* oil from the rind, were deduced as follows:

	Caprylic acid.	Capric acid.	Lauric acid.	Myristic acid.	Palmitic acid.	Stearic acid.	Oleic acid.	Linolic acid.
"Parings" oil, per cent.	2(?)	2	28	22	12	1(?)	23	10
"Kernel" oil, per cent.	9·5	4·5	51·0	18·5	7·5	3·0(?)	5·0	1·0

The evidence for the presence of acids of lower carbon content than caprylic acid, even after fractional distillation of the barium salts, was small. D. G. H.

Colour Test for Cineol. E. J. Schorn. (*Pharm. J.*, 1925, **114**, 232–233.)—One c.c. of a 15 per cent. solution of ammonium molybdate in dilute nitric acid containing 5 per cent. of ammonium sulphate, added to varying amounts of 10 per cent. solutions of cineol in alcohol gave blue colorations, and, on comparing equal volumes in Nessler tubes, the colours were found to be proportional to the amount of cineol present. D. G. H.

Iron in Tragacanth. G. Elliott. (*Pharm. J.*, 1925, **114**, 234–235.)—Nine samples of tragacanth were found to contain from 0·0015 to 0·01 per cent. of iron. When tragacanth is made up with salicylic acid as preservative a reddish-violet colour may therefore develop. D. G. H.

Detection of Ethyl Phthalate in Ethyl Alcohol. J. T. Raleigh and S. U. Marie. (*J. Amer. Chem. Soc.*, 1925, **47**, 589–590.)—The U.S. Government official test for the detection of ethyl phthalate in ethyl alcohol, depending on fusion with sodium hydroxide and treatment with resorcinol to form fluorescein, is found to be unreliable, as it gives a distinct fluorescence in absence of ethyl phthalate. The following test, although not conclusive, gives strongly indicative results:—One hundred c.c. of the alcohol are evaporated to small bulk and then poured into a large volume of water containing a few c.c. of sodium hydroxide solution; a milky cloud of ethyl phthalate will develop if this denaturant is present. The boiling point of the ester (290–297° C.) serves as a confirmatory test.

T. H. P.

The Value of the D.M. Index for Arsenobenzenes. A. Valeur and L. Launoy. (*J. Pharm. Chim.*, 1925, **1**, 4–22.)—The generally adopted limit of 19 to 21 per cent. for arsenic in arsenobenzenes is probably correct, but the ratio of arsenic to nitrogen is not of much value as a criterion of the product. The D.M. index itself (ANALYST, 1924, **49**, 43) is an empirical figure, which lacks sharp chemical definition, and even when the exact conditions laid down are followed, variable results are obtained. The only satisfactory way of examining arsenobenzenes is by physiological methods. D. G. H.

Rapid Microchemical Identification of Novocaine. G. Denigès. (*J. Pharm. Chim.*, 1925, **1**, 32.)—A few particles of the solid are dissolved in a drop of water (in the case of the free base by addition of a trace of acetic acid), and an equal volume of 5 per cent. sodium perchlorate solution is introduced into the centre of the drop by means of a narrow glass tube. Novocaine perchlorate shows characteristic prismatic needles when viewed under the microscope, and the identification is confirmed by the precipitation of silver chloride by means of silver nitrate solution. D. G. H.

Constitution and Chemical Properties of the Alkaloids of the Calabar Bean. E. Cattelain. (*J. Pharm. Chim.*, 1925, **1**, 59–69.)—Max and Polonovski, working on eserine (phytostigmine) discovered its oxide, geneserine, and have established the fact that these substances are the methyl urethanes of the nitrogen bases eseroline and geneseroline. They contain one benzene and two heterocyclic nuclei. Geneserine is formed by oxidation of eserine and is readily reduced again, the oxygen being directly linked to the pentavalent nitrogen atom, as in oxamines. In the plant it is formed from eserine by enzymic oxidation. Eseroline and geneseroline form two open chain nitrogen bases as degradation products, *viz.* eserolene (of which the ethoxyl compound is the only known derivative) and ψ-geneserolene. D. G. H.

Therapeutic Value of Extract of Ergot of Rye. A. Goris and A. Liot. (*Bull. Sci. Pharmacol.*, 1924, **31**, 379–390; *J. Pharm. Chim.*, 1925, **1**, 123–125.)—Aqueous extraction of powdered ergot of rye only withdraws a small proportion of the specific alkaloids, which is not appreciably increased by the addition of tartaric acid, the extract being naturally acid owing to the presence of hydrogen potassium phosphate. Further evaporation of the aqueous extract causes a certain amount of decomposition of the ergotinine, so that a powder containing 0·167 per cent. of alkaloid gave an extract containing only 0·242 per cent. of alkaloid, whereas it should theoretically have contained 1·2 per cent. The therapeutic action is due not only to ergotinine and other alkaloid bases, but also to amino bases formed during the preparation of the extract. D. G. H.

Detection of *Atractylis gummifera* in Liquorice Extract. P. Bertolo. (*Giorn. Chim. Ind. Appl.*, 1925, **7**, 12–14.)—The author maintains the superiority of his method over that proposed by Giuffrè (ANALYST, 1923, **48**, 30; 1924, **49**, 534), which gives uncertain indications. T. H. P.

Biochemical, Bacteriological, etc.

The Nitrogenous Groups of Plant Nucleic Acid. W. Jones and M. E. Perkins. (*J. Biol. Chem.*, 1925, 62, 557–564.)—When yeast nucleic acid is treated with dilute sodium hydroxide at room temperature it is decomposed into its nucleotides without the formation of any free phosphoric acid or free purine bases, and deaminisation does not occur. Experiments carried out by the authors are described, and tables of the results are given. Among the products three of the hitherto accredited nucleotides of yeast nucleic acid (guanine nucleotide, adenine nucleotide and cytosine nucleotide) can easily be isolated in quantity, but the fourth nucleotide (uracil nucleotide) cannot be found. This is especially remarkable because, of the four nucleotides, uracil nucleotide is the one least likely to be lost in analysis, on account of the great insolubility of its brucine salt in 35 per cent. alcohol. A large and unexpected amount of cytosine nucleotide is produced. The conclusion seems obvious: that the oxy-pyrimidine derivatives (uracil, uracil nucleoside and uracil nucleotide), which have hitherto been obtained from yeast nucleic acid, are not referable to an oxy-pyrimidine group in nucleic acid, but are secondary products formed during hydrolysis by deaminisation of the corresponding cytosine derivatives or their precursors. P. H. P.

Studies on Enzyme Action. XXX. Comparative Study of Characteristic Lipase Actions of Tissues of Different Animals and of some Human Tissues. K. G. Falk, H. M. Noyes and K. Sugiura. (*J. Biol. Chem.*, 1925, 62, 697–709.)—The average "pictures" of the lipase actions on ten esters of nine tissues of the rat, rabbit, ox, man, and dog (one tissue) are compared in a series of figures. The study of the type of action or "picture" of the ester-hydrolysing actions of a given tissue of an animal together with the absolute actions, both under standard conditions, may be taken to be characteristic in most cases. Definite similarities and differences are pointed out. It is impossible to state definite rules as to the magnitudes of the actions for the same tissue in different animals because of the differences in relative actions on the various esters. As a rule, the rat tissues are the most active, followed by rabbit, beef and human tissues in the order named. This applies to the special experimental conditions used and only in the most general terms. As for the absolute actions of the various tissues of the different animals, the only general statements which may be given point to the facts that the brain and muscle tissues show the smallest actions. A discussion of the results is given. P. H. P.

Fat-Soluble Vitamins. XXI. Observations Bearing on the Alleged Induction of Growth-Promoting Properties in Air by Irradiation with Ultra-Violet Light. E. M. Nelson and H. Steenbock. (*J. Biol. Chem.*, 1925, 62, 575–593.)—The work carried out by the authors is described fully, and charts and tables show the results. Confirmatory evidence is offered that irradiated rats put in the same cage with non-irradiated rats are able to induce growth in the latter, even when kept on screens to minimise consumption of excreta. Evidence is also presented that mere irradiation of the false screen bottoms of rat cages is sufficient to induce normal growth in rats when they are kept on a ration deficient in the antirachitic vitamin. No evidence was obtained that the above-mentioned reactions were brought about by secondary radiations acting externally upon the animals, nor that irradiation with ultra-violet light will confer growth-promoting or antirachitic properties on air. It is suggested that the antirachitic growth-promoting reaction was induced in the animals by the ingestion of photochemically activated constituents of the excreta. P. H. P.

Germicidal Action of different H-ion Concentrations on Bacteria. S. Sierakowski and F. Milejkowska. (*Compt. Rend., Soc. Biol.*, 1924, **91**, 714–715; *Chem. Abst.*, 1925, **19**, 90–91.)—The action of H-ions on micro-organisms is very rapid, and is increased by raising the temperature. By changing the temperature from 20° C. (?) to 45° C. the P_H at which typhoid bacilli are killed shifts from 4·2 to 5·1. Some of the organisms tested are given in the following list:

Species.	P_H range of tolerance.	Species.	P_H range of tolerance.
B. coli	3·8–10·02	*B. rhinosclerome*	4·4–10·02
B. typhoid	4·2–10·02	*B. ozene*	4·8–10·95
B. paratyphoid A	4·0–10·45	*B. Friedlander*	3·8–10·02
B. paratyphoid B	3·8–10·02	*St. aureus*	3·9–12·2
B. paratyphoid C	4·1–10·95	*St. citreus*	4·1–11·92
B. shiga	4·2– 9·63	*B. anthrax*	1·7–13·0
B. Flexner	4·6– 9·63	*B. diphtheria*	4·9–10·0
B. cholera	5·3–10·45	*B. pseudodiphtheria*	5·3–11·31

Measurement of Chemical Action in Micro-organisms, especially in Yeast. T. Bokorny. (*Allg. Brauer. u. Hopfenzeit.*, 1924, 7–9; *Chem. Abst.*, 1924, **18**, 3409.)—Experiments have shown that a definite amount of poison is required for a definite amount of living cell substance, and that the poison and living substance combine chemically in proportionate amounts. No cases were observed where a combination of poison and cells did not occur. The combination with the poison varies greatly with the amount, and especially with the active concentration of the poison, according to the kind of bacteria or yeast. It is recommended that in practice both the lethal dose and the activity of different dilutions of a disinfectant should be indicated. For example, in the case of formalin: lethal dose for 10 grms. of pressed yeast containing 25 per cent. of dry substance $= x$ grms.; active dilution from 7 per cent. upwards; the velocity of the reaction varies with the degree of the dilution, and the poisonous action increases with increase of temperature.

Determination of Lecithin in Blood. A. Grigaut. (*J. Pharm. Chim.*, 1925, **1**, 97–104.)—The determination is made by means of a colour reaction based on the fact that phosphomolybdic acid in the presence of suitable reducing agents gives a blue colour, and that, under certain conditions, the excess of molybdic acid is without effect on the coloration. One c.c. of blood serum is poured, drop by drop, with shaking, into 40–50 c.c. of a mixture of 3 parts of 95 per cent. alcohol and 1 part of ether, and, after 10 minutes, filtered through an ashless paper which is then washed with further portions of ether. The united extracts are evaporated just to dryness, and the residue taken up while still warm with dry ether, filtered or centrifuged, the paper washed, and the ethereal solution transferred to a Kjeldahl flask and evaporated to dryness. A few c.c. of distilled water, 1 c.c. of concentrated nitric acid, and 1 c.c. of sulphuric acid (sp. gr. 1·843) and a glass bead are added in succession, and the mixture boiled. As soon as the water has disappeared and when fumes appear, the flame is lowered and heating continued until the solution is colourless. After cooling, 30 c.c. of distilled water are added and boiled down to 10 c.c. (to ensure pyro- and meta-phosphates being transformed to orthophosphates), and the contents are then transferred to a 50 c.c. graduated flask, made up to about 40 c.c. and neutralised with 40 per cent. sodium hydroxide solution. Five c.c. of the standard solution of phosphate (containing 0·044369 grm. per litre of $NH_4H_2PO_4$; equivalent to 1·5 mgrm. of lecithin in 5 c.c.) and the same volume of sodium hydroxide solution as was used above, are put into another 50 c.c. flask, and neutralised with dilute sulphuric acid. The cooled solutions are each made up to 50 c.c., and 2 c.c. of the sulpho-molybdic reagent (containing equal parts of 10 per cent. ammonium molybdate and sulphuric acid of sp. gr. 1·843) 0·5 c.c. of the stannous reagent (0·1 grm. of tin foil dissolved in 2 c.c. of concentrated hydrochloric acid and 10 c.c. of distilled water) are added. After 2 minutes the colours are compared in a Duboscq colorimeter. Then the percentage of lecithin in the serum is $1{\cdot}5 \times E/S$ grms., where E is the depth of the type solution and S the depth at which the solution under examination shows the same tint. D. G. H.

Method for Determining Methæmoglobin in the Presence of its Cleavage Products. J. B. Conant and L. F. Fieser. (*J. Biol. Chem.*, 1925, **62**, 623–631.)—As a spectrophotometer is not always available, a method has been developed for determining methæmoglobin in the presence of its cleavage products or other coloured material. It consists in measuring the increase in oxygen capacity, resulting from the action of a powerful reducing agent which quantitatively converts the methæmoglobin to haemoglobin. Sodium anthrahydroquinone-β-sulphonate is a satisfactory reducing agent for this purpose, although the oxygen capacity after reduction must be determined rapidly, and the equilibration with pure oxygen must be of very short duration. Disodium anthra-hydroquinone-2,6-disulphonate is a somewhat less satisfactory reagent whilst sodium hyposulphite cannot be used at all because its oxidation products catalyse so greatly the re-formation of methæmoglobin. In the calculation one really measures the amount of material which by itself has no oxygen capacity, but which, after treatment with a reducing agent, combines with oxygen reversibly. As far as is known, the only substance which has this very peculiar property is methæmoglobin. P. H. P.

Effect of Sodium Benzoate Ingestion upon the Composition of the Blood and Urine, with especial Reference to the Possible Synthesis of Glycine in the Body. W. W. Swanson. (*J. Biol. Chem.*, 1925, **62**, 565–573.)—Experiments, described in detail, were made to obtain values for the non-protein nitrogen constituents in the blood and urine found by simultaneous analyses after ingestion of sodium benzoate, and to investigate what bearing, if any, these values may have on the synthetic ability of the human body to produce glycine for conjugation with the benzoic acid. Analyses show:—(1) No increase in the urea nitrogen content of the whole blood and plasma, but a decrease in the urea nitrogen content of the urine; (2) an increase of the uric acid content of the whole blood and plasma, and a marked decrease in the uric acid content of the urine; (3) the glycine portion of the blood amino acids and of those which are normally excreted while fasting appears to be only a small percentage, if any, of the total; and (4) the experimental findings suggest a probable synthesis of glycine from the constituents which normally are converted into urea, but it appears improbable that uric acid supplies any glycine. The results are discussed. P. H. P.

Detection of Amino-nitrophenols in Urine. L. Desvergnes. (*J. Pharm. Chim.*, 1925, **1**, 56–59.)—The Derrien azo reaction cannot be applied directly to the urines of persons absorbing iodine or iodised peptones, owing to the masking of the colour developed, and it is necessary in such cases to add 2 drops of sulphuric acid to 5 c.c. of urine, extract three times with chloroform, filter and then proceed with the test. To 10 c.c. of the filtrate are added 1·5 c.c. of a 0·5 per cent. sodium nitrite solution, and, after 10 minutes' contact in very cold water, the mixture is poured into 1 c.c. of a freshly prepared solution of 0·5 per cent. ammoniacal β-naphthol. After a few minutes 10 c.c. of ether are added, and the mixture gently shaken, after which a pale pink to red-violet colour indicates the presence of amino nitrophenols in the urine. D. G. H.

Toxicological and Forensic.

The Arsenic Content of Baking Powder. S. Goy and A. Köhler. (*Chem. Zeit.*, 1925, **49**, 189.)—In consequence of a case of arsenical poisoning in Breslau, traced to the use of potassium carbonate as baking powder, a series of 68 samples from that town and province was examined, and 12 were found to contain arsenic ranging in amount from 0·09 per cent. to 0·38 per cent. as As_2O_5 (no arsenic was present in the arsenious condition). This amount would be poisonous if the potassium carbonate were used as a baking powder. The arsenic originates in the arsenical dips used for sheep, from the wool of which the potassium carbonate is prepared. It is also pointed out that fellmonger's wool coming from skins which have been unhaired (de-wooled) with lime and arsenic paints may be another source of arsenic contamination. Of 133 baking powders not made with potassium carbonate, none was found to contain arsenic. R. F. I.

Toxic Action of Commercial Ether. E. Remy and F. Neuhart. (*Zeitsch. Unters. Nahr. Genussm.*, 1924, 48, 445–449.)—During investigation of a cocoa alleged to have caused sickness, physiological experiments were made by injection of some of the ether-extracted fat and of aqueous extracts into mice. The ether extract in each case caused poisoning, but not so the aqueous extracts. It was found that the ether left a residue on evaporation amounting to 0·03 per cent., which contained dioxyethyl-peroxide and vinyl alcohol, both of which were proved to be highly toxic. The vinyl alcohol may be detected by shaking 50 c.c. of the ether with 9 c.c. of potassium bicarbonate solution and 20 c.c. of mercuric chloride solution, when a white amorphous precipitate of $Hg_3Cl_2O_2C_2H_3$ is given in the presence of this alcohol. Dioxyethyl-peroxide is detected by means of potassium iodide. It is evidently essential to employ only the purest ether for extractions which are to be used for physiological experiments. H. E. C.

Organic Analysis.

Rapid Method for Determining Carbon in Organic Compounds. H. D. Wilde (jun.) and H. L. Lochte. (*J. Amer. Chem. Soc.*, 1924, 47, 440–446.)—The compound is burnt in presence of compressed oxygen and of standard barium hydroxide solution (0·5 *N*) in the calorimetric bomb, the carbon dioxide thus formed being absorbed by the alkali when the bomb is subsequently shaken. The solution is then titrated with 0·5 *N* hydrochloric acid in presence first of phenolphthalein and then of methyl orange, the amount of acid required between the two end-points being equivalent to the carbon dioxide formed. If the operations are carried out rapidly in a well ventilated laboratory, the carbon dioxide of the air may be neglected. Solid substances should be compressed into pellet form before weighing and, if difficult to burn completely, should be placed in an ordinary gelatin capsule, this being previously weighed. Such capsules, which are hygroscopic and should be stored in a desiccator, are of uniform carbon content, so that the necessary correction is readily determined. Liquids may be weighed in the capsules except when these are rapidly attacked; in such case one of the regular methods of burning liquids for calorimetric purposes must be used. If the compound contains sulphur, this may be determined gravimetrically in the titrated solution. Nitrogen from nitrogenated compounds does not interfere with the carbon determination and seems to escape almost entirely as free nitrogen.

Compounds containing halogens, however, require special treatment, oxyhalogen compounds, present in the alkali after the combustion, being destroyed by a reducing agent, such as hydrazine hydrate, the solution being then acidified and boiled, and the carbon dioxide thus liberated reabsorbed by barium hydroxide before titration.

The above procedure gives excellent results with compounds difficult or impossible to burn completely by the ordinary combustion method. T. H. P.

Application of Sodium Hyposulphite (Hydrosulphite) in Organic Analysis. E. Clark. (*Chem. News*, 1925, **130**, 132.)—In establishing the benzenoid nature of a compound by the usual method the very bulky precipitate of zinc hydroxide may altogether mask the formation of the red hydroxy-azo compound. Instead of using zinc and hydrochloric acid as the reducing agent for the nitro-body, the author has found great advantage in using sodium hyposulphite in alkaline solution. The supposed benzenoid compound is nitrated in a test-tube as usual, and the product cooled and made alkaline with sodium hydroxide. To the alkaline solution is added a small amount of solid sodium hyposulphite, with shaking. The mixture is then heated to boiling, acidified with excess of hydrochloric acid, and boiled till all smell of sulphur dioxide has disappeared. The solution is then cooled, treated with a few drops of sodium nitrite solution and added to alkaline naphtholate solution, when a brilliant red colour results if the original substance was of aromatic character. Nitro-compound—> sulphonic derivative of the amino compound—> amine hydrochloride —> diazonium compound—> red hydroxyazo compound. R. F. I.

Occurrence of Free Pentoses in Plants. Effect of Extraction of the Sugars with Ammoniacal Alcohol. D. T. Englis and C. Hale. (*J. Amer. Chem. Soc.*, 1924, **47**, 446–449.)—After subjection to prolonged heating with ammoniacal alcohol in a reflux apparatus and to considerable concentration under reduced pressure, an alcoholic solution containing sucrose and invert sugar appears to contain appreciable amounts of pentoses when tested by either the Krober-Tollens or the fermentation method. In absence of ammonia the above treatment gives no apparent pentose according to the fermentation method, but a small quantity by the other method of testing. The amounts of pentose found are not sufficient to account for the total quantities reported in plant extracts, but it is evident that any method of extracting sugars which involves the use of ammonia and, possibly, other weak alkali, should be avoided or used with caution.

T. H. P.

Inorganic Analysis.

Volumetric Determination of Pb_3O_4 in Red Lead. E. F. Figg. (*J. Soc. Chem. Ind.*, 1925, **44**, 68T.)—To 1 grm. of red lead in a glass mortar are added 40 c.c. of a saturated solution of sodium acetate in 5 per cent. acetic acid, and a known excess (40–50 c.c.) of 0·1 *N* sodium thiosulphate solution. Rubbing is carried on until all undecomposed red lead has disappeared, and the addition of a few c.c. of 5 per cent. potassium iodide solution hastens the reaction. Starch solution is added, and the excess of thiosulphate solution is then determined by titration with 0·1 *N* iodine solution. The excess of sodium acetate prevents precipitation of lead iodide, and the end-point is sharp. D. G. H.

The Alloxantin Reaction for Ferric Iron. G. Denigès. (*Compt. Rend.*, 1925, **180**, 519–520.)—Alloxantin forms an exceedingly delicate reagent for ferric iron when present as a complex ion, such as citric and tartaric compounds, which do not react with the ordinary reagents. The reagent should be freshly prepared by dissolving 0·1 grm. of alloxantin in 10 c.c. of *N* sodium hydroxide solution. If the solution has a pink colour, it should be boiled until the colour just disappears, then cooled. The reagent is now added to the solution containing the iron, and a blue colour is obtained in the presence of 1 mgrm. of iron per litre. By using a larger volume of solution the reaction can be made much more delicate. The presence of alkalis or organic acids does not interfere. H. E. C.

Separation of Zinc and Nickel by Hydrogen Sulphide. A. Kling and A. Lassieur. (*Compt. Rend.*, 1925, **180**, 517–519.)—By careful attention to the acidity it is possible to precipitate zinc sulphide alone in the presence of nickel. The solution must be acidified with acetic acid and controlled by ammonium salts. Hydrogen sulphide must be boiled off before the P_H is determined, as it interferes both with a hydrogen electrode and with indicators. It is found that nickel is completely precipitated only when the P_H lies between 2·8 and complete neutrality. Zinc is precipitated in 45 minutes when the P_H is at 2·4. The presence of salts disturbs these values. H. E. C.

Electrolytic Determination of Zinc in Alloys. J. W. Springer. (*Zeitsch. anal. Chem.*, 1925, **65**, 315–317.)—One grm. of brass or bronze is dissolved in strong nitric acid, the solution diluted slightly and treated with enough ammonia to redissolve the precipitate. The solution is filtered, if necessary, and the filtrate neutralised with sulphuric acid (1:1) and treated with 10 c.c. excess. The copper is deposited from the boiling solution by a current of 4 amp.; the electrolyte is neutralised with sodium hydroxide, of which an excess of 10 grms. is added. The solution is electrolysed at a temperature near the boiling-point with a current of 4 amps. for 15 to 20 minutes, the platinum wire anode rotating at 800–1000 R.P.M. The amalgamated brass gauze cathode is washed without interruption of the current, rinsed with alcohol, and dried at 70° to 80° C. The results are stated to be good (*cf.* ANALYST, 1924, **49**, 403). W. R. S.

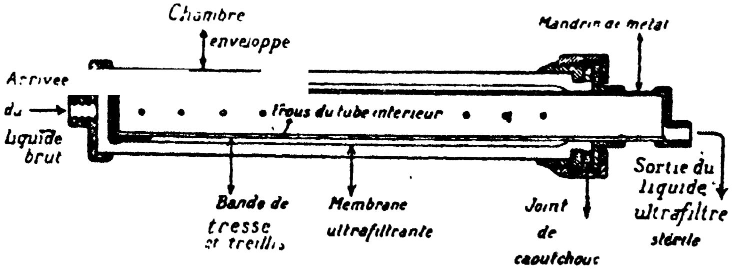
Elimination of Phosphoric Acid in Qualitative Analysis by Bismuth Subnitrate. A. Keschan. (*Zeitsch. anal. Chem.*, 1925, **65**, 346–351.)—The filtrate from the hydrogen sulphide precipitate is evaporated almost to dryness and again evaporated to small bulk with nitric acid to eliminate most of the hydrochloric acid. If iron is present, the solution is diluted with 10 to 15 volumes of water, saturated with hydrogen sulphide, treated with excess of solid bismuth subnitrate, boiled and diluted with an equal volume of hydrogen sulphide water. It is left to cool somewhat and filtered warm. Iron in the ferric condition would prevent the complete precipitation of the phosphoric acid through formation of complex anions; the hydrogen sulphide is added as reducing agent. A certain amount of bismuth sulphide is formed, but this is mostly converted into nitrate in the boiling liquid. The precipitate, which is removed by filtration, should always be discoloured by sulphide, which shows that the iron remains reduced; if necessary, more hydrogen sulphide water must be added. The iron may also be reduced by hydrazine hydrochloride. If iron is absent, the required excess of bismuth subnitrate is at once added to the concentrated nitrate solution after evaporation; the mass is then diluted gradually with 20 to 30 volumes of water and boiled until a small filtered portion gives no reaction with molybdate mixture. If the reaction persists after 15 to 30 minutes, more subnitrate must be added. W. R. S.

Physical Methods, Apparatus, etc.

Spectrophotometric Determination of P_H. F. Vlès. (*Compt. Rend.*, 1925, **180**, 584-586.)—Spectrophotometric methods of determining P_H (*cf.* Holmes, ANALYST, 1924, **49**, 253) suffer from the disadvantage that the absorption of the indicator, colog (I/I_0), is a function of two independent variables, namely P_H and the concentration of the indicator, so that an awkward readjustment becomes necessary on every occasion when there is divergence from the standard experimental conditions. For indicators composed of two tautomeric spectral components and for values of P_H which are not extremely low, it is shown that the concentration of the hydrogen ions may be expressed as a function of the ratio of the absorptions for two different wave lengths, and independently of the concentration of the indicator, preliminary empirical standardisation being unnecessary. The relation found is applicable in practice to some indicators, but with others, such as crystal violet, anomalous behaviour is observed owing to the transformation, between the values 3 and 1 of P_H, of the violet form (λ592) into the green form (λ632), which then undergoes progressive destruction to give a yellowish form.

T. H. P.

A Collodion Membrane Ultra-filter. Physico-Chemical Analysis of Solutions. E. Fouard. (*Ann. Chim. anal.*, 1925, **7**, 33-34.)—The apparatus shown in the figure has been devised so that a collodion membrane can be used for ultrafiltration in such a way that it cannot lose its shape and can withstand pressures (up to 40 atomos.) without any alteration in its properties. The apparatus consists of a rigid body, an enamelled metal tubular mandrel, protected against different reagents, and with small holes evenly distributed over its surface; this is bound with an artificial silk braid and over it is fixed a lattice-work of



strong thread with a large mesh of 1 or 2 mm. The whole is closed at one end and open at the other. By a special technique, a covering of collodion, which does not penetrate the envelope of braid, but completely immerses the meshes of the network of thread, is placed on the system, thus, by the coagulating effect of the water, forming a membrane (which is no longer fragile but strong owing to the mesh) all over the surface. The whole apparatus is placed in a metal case, so that it can be fixed in a pressure chamber. Ultra-filtration takes place from the exterior to the interior of the mandrel. This apparatus can be used regularly for a year for sterilising water without needing attention. P. H. P.

New Method for Examining the Interior of Pearls. B. Szilard. (*Compt. Rend.*, 1925, **180**, 433–436.)—This method, which is applicable to pearls, bones, fibres, wood, etc., consists in examining the object when completely immersed in a liquid of suitable refractive index and strongly illuminated from below. The liquid serves to prevent the reflection of the light from the surface, so that the rays actually penetrate the object. Over an electric lamp is placed a flat cell containing water (to keep the condenser cool), then an optical condenser, and then the pearl immersed in cedar-wood oil; over this is a diaphragm and finally the low-power lens of a microscope. In the case of pearls it is distinctive that in cultivated pearls there are visible, in the interior, concentric striations corresponding to the layers formed by the deposition of the mother-of-pearl. In genuine natural pearls these striations are absent. Diagrams and photographs are given.

H. E. C.

ERRATUM.

Hydrogen Ion Concentration of Natural Waters. ANALYST 1923, **48**, 397, line 3: *for* "$P_H = 6{\cdot}3$" *read* "$P_H = 8{\cdot}3$."

Reviews.

A SYSTEM OF PHYSICAL CHEMISTRY. Vol. III. QUANTUM THEORY (Third Edition). By WILLIAM C. MC. C. LEWIS. Pp. x.+410 (with 24 figures). London: Longmans, Green & Co. 1924. Price 15s.

The application of the quantum mechanism to the elucidation of the phenomena attending the interconversion of atomic and radiant energies has led us to the conclusion that the structures of atoms change in a discontinuous manner as their energy content increases. Thus it would appear that the individual atoms of a mono-atomic gas differ from one another, not only in respect of their velocities of translation, but also in regard to their internal structures. The extension of this view to molecules introduces a conception into chemistry the influence of which will undoubtedly be comparable with that of Dalton's atomic theory, over a hundred years ago.

Physical chemists have been aware, almost from the beginnings of the quantum theory, of its importance for the study of the kinetics of chemical change, and Professor Lewis was one of the first to advocate its application to chemistry. On the other hand, the majority of chemists do not appear to have grasped its significance for the interpretation of the constitution of chemical compounds. Deductions as to constitution based on chemical behaviour are very frequently at variance with those arrived at from a study of physical properties, for chemical behaviour is determined by the constitution of an extremely small fraction of the total number of molecules present, namely, those in a specially active state, whereas all of the molecules contribute their quota in any physical measurement. The application of quantum considerations should throw light, not only on the active, but also on the other molecular states present in a chemical system.

Professor Lewis's book has already played a great part in the development of the more recent views of chemical behaviour, and the new enlarged edition is very welcome. The present edition is twice the size of that issued in 1919, and this increase in size is an indication of the rapid rate of development of the subject covered by the volume. It has been entirely re-written, except for the first few chapters, which have been thoroughly revised. The increase in size is partly due, however, to eight appendices at the end of the book. The paper on which it is printed is a great improvement on that of the 1919 edition, and the publishers are to be congratulated on the low price at which the work is issued.

The scope of the book is very wide and includes accounts of nearly all of the recent physical investigations which have, or are likely to have, any bearing on chemistry. The selection of the material from the original papers and its arrangement in the form of a coherent argument has been a difficult task, for it is not always possible to correlate work done in isolated fields. In the first two chapters, which deal with the interchange of energy between resonators and radiation, it is shown how the failure of the equipartition principle led to the introduction by Planck of the quantum theory. This is then applied to the energy contents of solids and gases, the equations of Einstein, Nernst-Lindemann, and Debye being given. A very interesting summary on mono- and di-atomic gases and of the degradation of gases from the point of view of the quantum theory is included. In the chapter on atomic and molecular structure, the Bjerrum and Kratzer theories of infra-red band spectra and the various atomic models, due to Bohr, Langmuir, Whittaker, etc., are points of special interest. The work of Henri and others on the molecular structure of organic compounds, which should have appeared in this place, has been ignored, and the space afforded to the orbits of the electrons in the chemical bond (three pages) appears to be unduly small, considering the importance of the question.

The application of the quantum theory to volatilisation, sublimation, and fusion, the thermionic and photo-electric effects, and ionisation and resonance potentials receive their due attention. Chemical reactivity, both thermal and photochemical, is treated very fully, largely from the point of view of the radiation hypothesis, in regard to which the author has summarised the evidence both for and against. The author's development of this section is open to criticism, in that he takes an extreme view of chemical reactivity; he has, on the strength of the difficulties inherent in explanations of the mechanism of the hypothetical monomolecular reaction, eliminated kinetic energy of translation from his considerations. Even if chemical change is limited to those collisions occurring between active molecules, it is not certain that the speeds on collision of all of these are sufficient for the penetration of the first line of defence, the outer envelope of the molecule. A minimum kinetic energy would appear to be necessary which, it is true, may be so low in a number of reactions that all collisions between active molecules are fruitful, and the A in the Arrhenius equation equals $h\nu$, where ν is the frequency of the radiation activating the molecule. In the majority of the reactions studied, however, this is certainly not true.

The energetics of crystal structure, including the affinity of atoms for electrons, and atoms for atoms, is dealt with in Chapter VIII., and a short account of molecular and atomic forces is given. The bearing of the quantum theory on the Nernst heat theorem, chemical constants, and mass action equilibria forms the last chapter.

Appendices on special points not referred to in detail in the text are given at the end of the volume, including Maxwell's distribution law, the quantum theory, and the correspondence principle by James Rice, on the Sommerfeld-Bohr theory by A. McKeown, and on Einstein's law of the photochemical equivalent by R. O. Griffith.

The volume contains in a small compass an account of the modern developments based on the quantum theory, and as a book of reference will prove of great service. It is essentially a monograph, not a text book in the true sense of the word. It is to be doubted if the time is yet ripe for a text book on this subject, especially as the body of knowledge within its purview is undergoing such rapid changes. W. E. GARNER.

L'ORIGINE TOURBILLONNAIRE DE L'ATOME ET SES CONSÉQUENCES. PAR JEAN VARIN D'AINVELLE. Paris: Gauthier-Villars et Cie. Pp. 214. Price 10 fr.

The vortical origin of the atom, if indeed its origin be vortical, has attracted but little attention in the present century. Physicists and chemists have devoted their energies rather to the study of the actual and ascertainable properties of atoms and their constituents than to speculations as to their origin. The author of this book has revived a theory which may recall to some readers the Helmholtz Memorial Lecture given by Fitzgerald in 1896. He said: "To the Chemical Society the question of most importance is how Helmholtz's work bears on chemical theories. Unfortunately, there is very little really known about this in respect of vortex motion. Lord Kelvin has made the brilliant suggestion that atoms are vortex rings, and upon this all subsequent guesses have been founded; for our knowledge in this direction is very little better than guesses. Helmholtz had shown that vortices in a perfect liquid could neither create nor destroy one another. Once created, a vortex ring is permanent like an atom of matter." On this "correspondence principle" the vortical theory of the nature of atoms is based.

Clearly atoms must have had some origin. The ether is a convenient conception furnishing a means of evading the difficulties of action at a distance and, as the late Lord Salisbury said to the British Association, providing a nominative to the verb "to oscillate." Vortices in such a fluid would have all the permanence attributed to atoms and would differ from the circumambient ether only by reason of their motion and not qualitatively. A merely dynamical origin of matter is thus made possible, and we have a homogeneous etherous universe.

The vortical theory is not without its difficulties. Fitzgerald pointed out that it is difficult to fit in the constancy of the relation of gravitation and inertia at varying temperatures with the vortex theory. This and other difficulties led Fitzgerald to "prefer in our ignorance the horn of the dilemna, that holds that atoms are not simple thin vortex rings in an otherwise unmoving liquid."

M. Varin D'Ainvelle says: "Je vais essayer dans cet ouvrage de déduire les lois de la Physique de l'hypothèse suivante: les atomes sont des tubes de tourbillon nés et se mouvant dans l'ether, fluide auquel nous n'attributons aucune propriété spéciale comme le serait une rigidité anormale, mais qui ressemble aux fluides réels ordinaires, étant comme eux visqueux, compressible et sujet à des changements de température. Il est seulement plus subtil que n'importe quel fluide connu, en entendant simplement par là que ses molécules sont fort petites et tres peu denses. Les qualités énumérées plus haut ont un ordre de grandeur en rapport avec cette subtilité. On peut dire que l'ether ainsi conçu répond à la conception intuitive que nous nous en faisons."

The Bohr or Langmuir atom is a gross and all too simple structure compared with our author's "tourbillon E" or hydrogen atom. Brought into being by the encounter of two currents of ether, the atoms of the universe started as thin vortex rings which on rupture became hollow cylinders, and will finally, like those larger vortices, the heavenly bodies, become solid spheroids. The evolution is naturally slow, and our atoms are at present hollow cylinders with which are associated a Newtonian attraction originating from the contraction vector giving rise to *mass*, *magnetism* due to the vector of translation, and *electricity* due to the vortex vector. "Nous pouvons donc donner cette définition de la matière: c'est la portion de volume de l'ether dont chaque molécule est chargée d'une quantité de mass, d'une quantité de magnetisme, et d'une quantité de l'électricité: ces quantités, toujours les mêmes et indépendantes les unes des autres, sont des constantes de la nature. Les propriétés qui en découlent sont additives. La réunion d'un certain nombre de telles molécules constitue un atome de matière ou tourbillon E. Ce nombre est constant, mais non rigoureusement. Il subit des fluctuations autour d'une valeur moyenne."

Having endowed his atoms with their necessary properties, the author finds no difficulty in accounting for those of matter. He strays occasionally into cosmogony and metaphysics. It is all very clever, but it is difficult to feel that it has any relation to that which is. The atom and the electron may be said to be real. The quantum has been a fertile and necessary conception. Hard particles may be, as Fitzgerald said, "an abomination." If one could only think of the harmless necessary* ether as a continuum all would be well—except perhaps many theories. The necessity of giving it convenient properties has led to a jelly, a foam, and a lattice structure being ascribed to ether. The author considers it as a series of fluids made up of molecules, each fluid being finer grained than that which is buoyed in it. This seems to give an unending series of hard particles. One is reminded of William of Occum's Razor, "Entia non sunt multiplicanda praeter necessitatem."

For one modern concept M. Varin D'Ainvelle has no use, the positive nucleus. His "tourbillons T" or electrons serve all his purpose. The positive nucleus is none other than the atom deprived of its electron. For us negative electricity has the electron for its atom, and positive electricity does not exist. The positive ion is the atom deprived of an electron. This "one fluid theory," as it were, is made feasible by his very complicated atom.

As to Einstein's theory of relativity the author is neutral. So far he needs it not, but he has not found it necessary to repudiate it. He can take it when he feels disposed.

It is impossible to do justice to a book of this kind within the limits of an ordinary review. Those who are attracted will, perhaps, read it and devise tests for the theory propounded. One wonders are such books written from a feeling of the need of an explanation of phenomena or as rhapsodies in mathematical metaphysics. It may all be true, but, alas, how very complicated. One feels the profundity of the words which Oliver Heaviside, who died within the last few weeks, wrote years ago: "Even if we resolve all matter into one kind, that kind will need explaining. And so on, for ever and ever, deeper down into the pit at whose bottom truth lies, without ever reaching it."

J. H. Coste.

The Constituents of Coal Tar. By P. E. Spielman, Ph.D., F.I.C. Pp. xii. + 219. London: Longmans, Green & Co. 1924. Price 12s. 6d.

This volume is the latest in the interesting series of monographs on industrial chemistry edited by the late Sir Edward Thorpe.

The author's object of placing before those chemists interested in the utilisation of coal tar products, a volume containing a summary of the researches of the very large number of workers in this field is a very laudable one. Hitherto short chapters in comprehensive works, such as that of Lunge, have been the only source of information other than the laborious study of current literature. Recently a great deal of work has been carried out on tars of all kinds, particularly in Germany, and this volume, while it does not claim to be encyclopaedic, will serve as a practical guide and as a useful summary of the information available to 1924.

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* For arguments against the necessity of the ether see Dr. Jeans' Kelvin Lecture (*Nature*, March 7, 1925).

The volume is divided into five parts, the first of which is a general summary of the compounds which occur in tars of different kinds. The other four parts deal with these in their various classes of hydrocarbons, oxygen compounds, etc. The important compounds in each class are treated in detail, their occurrence commented upon, and their physical and chemical properties described at length.

In Part I. the author points out the relation between low and high temperature tars, but, unfortunately, makes no reference to the effect of the type of coal on the composition of the tar. This omission is continued throughout the book, and the reader is continually at fault regarding the nature of the coal used. In some cases, also, tars are described which are by no means representative. For example, some of the data in Table 1 are obviously incorrect (*e.g.* low temperature tar prepared by modern processes has a density of 1·05 not 1·13, and contains much less than 61·4 per cent. of pitch). Considerable amplification of Part I., with particular reference to the different tars available and their properties, would have made the volume much more valuable. In Parts II. to V. this could have been followed up by mentioning in the generalised statements the class of coal and the method of tar preparation. The information regarding the different chemical substances described is very well set out and extremely comprehensive but, again, the space devoted to their occurrence is very limited.

The bibliography at the end of the book is good, and its extent (525 pages) is an indication of the trouble which the author has taken to make available to his readers the origin of the many papers on this subject. In some cases where different authors have the same name it is a pity that they are not referred to separately in the index and their initials quoted. Certain errors have crept in as, for example, ref. 12 should read volume 105 instead 104.

The book is well written and printed and should be welcome in the libraries of gas and tar works. When the march of progress calls for a further edition it is to be hoped that the author will make considerable extensions in his treatment of the origin of the tars described and of the variations of compounds in each type.

J. G. KING.

METALLURGY. AN ELEMENTARY TEXT-BOOK. E. L. RHEAD, M.Sc.Tech., F.I.C., A.I.M.E. New Edition. Pp. xii.+403. London: Longmans, Green & Co. 1924. Price 7s. 6d.

The preceding edition of this work having gone through six new impressions, the text-matter has been brought up to date in the present edition by suitable additions and deletions. The book is so well known that a detailed analysis of its contents is unnecessary; in the writer's opinion the author is at his best in the chapters on general metallurgy, iron, steel, and copper, whilst his treatment of the non-ferrous metals (other than copper) and especially some minor metals is not uniformly happy. Mention, however brief, might have been made of such advances in metallurgical practice as the electrolytic refining of lead, silver, and gold, the combined amalgamation-cyanide process for silver ores practised in Canada, and the modern hydrometallurgy of copper. The metallurgy of aluminium is dismissed in rather less than six lines; this may have been considered adequate thirty years ago, but the present importance of aluminium and the peculiarity of its metallurgy make a more detailed treatment imperative in an up-to-date text-book, even though elementary. The uses of cobalt and its growing importance as an alloy metal are mentioned, but nothing is said about its extraction or occurrence.

A great fault the reviewer has to find with the book is the careless style in which too many passages are written. The following sentence, picked out at random, is a case in point: "In roasting blende containing much iron the solubility of the zinc is diminished by the formation of zinc ferrate" (p. 369). This may be taken to mean that all the zinc is converted into "ferrate," and that it is more difficult to dissolve zinc "ferrate" than the oxide; whereas the author evidently means that part of the zinc is converted into zinc ferrite, which is insoluble in acid, with the result that the recovery, not the solubility, of the zinc is diminished. Under Gold Parting (p. 344) is found the curious term "osm.-iridium," which can hardly be a misprint, as it occurs three times in the same paragraph. Platinum is alleged to occur (p. 380) "with the rare metals, rhodium, osmium, iridium, ruthenium, rubidium" (*sic.*). The terms "quick silber" (p. 287) and "braque" (p. 68) pass for German. Similar minor inaccuracies and misprints are fairly numerous, implying careless proof-reading.

On the whole, it may be said that the revision of the book has been too conservative; obsolete processes have been accorded a little too much space, whilst some modern processes of quite considerable importance have been neglected.

W. R. SCHOELLER.

Publications Received.

INDEXED LIST OF BRITISH STANDARD SPECIFICATIONS AND REPORTS (1925). British Engineering Standards Association. Price 1s. 2d. post free.

INTRODUCTION TO ORGANIC RESEARCH. By E. EMMET REED. Constable & Co. 1925. Price 24s. net.

YEAR BOOK OF SCIENTIFIC AND LEARNED SOCIETIES (1923–1924). Chas. Griffin & Co. Price 15s.

COLLECTIVE INDEX OF THE JOURNAL OF THE INSTITUTE OF BREWING, 1911–1923.

MAY, 1925. Vol. L., No. 590.

10 JUN.

THE ANALYST

PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held on Wednesday, 1st April, 1925, at the Chemical Society's Rooms, Burlington House. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

Certificates were read for the first time in favour of:—Messrs. Lewis Eynon, B.Sc. (Lond.), F.I.C., Jack Rowan Heather, Frederick George Hitchman, and William David Rogers, B.Sc. (Hons. Lond.), A.R.C.S., F.I.C.

Certificates were read for the second time in favour of:—Messrs. George William Fraser Holroyd, M.A. (Oxon.), F.I.C., Cecil Eric Keeley, Andrew Francis MacCulloch, M.A., B.Sc. (Edin.), A.I.C., Frank Vegetus North Mitchell, Charles Henry Thomson, and Walter Peter Whitley, B.A. (Oxon.).

The following were elected Members of the Society:—Messrs. Douglas James Talbot Bagnall, A.C.G.F.C., A.I.C., Reginald Henry Coysh, M.Sc., A.I.C., George Van Barneveld Gilmour, B.Sc., A.R.C.Sc.I., A.I.C., Percy May, D.Sc., F.I.C., John Parry, Wilfrid Smith, B.Sc., A.I.C., and Miss Winifred Wright, B.Sc., A.I.C.

The following papers were read:—"Quantitative Determination of Cotton, Linen and Wood Fibres in Paper Pulp," by W. Dickson, F.I.C. (under the Analytical Investigation Scheme); "Proposed Standards for Lemon Cheese," by G. D. Elsdon, B.Sc., F.I.C. (followed by a general discussion on the *Desirability of Standards for Food Products*); and A Demonstration of:—"Laboratory Filters made of Sintered Glass and their various Uses," by P. H. Prausnitz, Dr. Ing.

Obituary.

SIR EDWARD THORPE, C.B., F.R.S.

Thomas Edward Thorpe was born on December 8th, 1845, the son of a Manchester merchant; he died on February 23rd, 1925, at Salcombe in South Devon, where he had built a house and constructed a beautiful garden on the top of the steep hill overlooking the far reaches of the inlet of the sea. In the 80 years of his life he worked to within a few days of his death with a great breadth of outlook, contributing to the advancement of knowledge in many directions.

On leaving Owens College, Thorpe went to Heidelberg and Bonn, at the former place coming under the influence of Bunsen. His first appointment was as assistant to Roscoe, and their long friendship resulted in the record of the life of Roscoe which Thorpe has given us. Thorpe left Manchester to be Professor of Chemistry at the Andersonian Institution, Glasgow; thence in 1874 to the Chair of Chemistry in the Yorkshire College, Leeds (the laboratory of which he designed), and in 1885 to South Kensington. In 1894 he became Principal of the Government Laboratory, returning in 1910 to South Kensington to become Professor of Chemistry and Director of the Chemical Laboratories for two years, after which he retired to Devonshire. Thorpe married in 1870 the daughter of Dr. John Watts of Manchester, who survives him. He leaves no family.

It is not possible to do more than indicate briefly the wide field which the activities of Thorpe covered. In addition to his strenuous professorial work, he took part in several magnetic surveys, both in this country and abroad, and in four solar eclipse expeditions. The sea voyages connected with these may have led to that love for the sea which made him an expert and devoted yachtsman.

Honours came to him in many directions. In 1895 he was President of the Society of Chemical Industry, in 1899–1901 President of the Chemical Society, and so recently as 1921 of the British Association Meeting in Edinburgh. He received honorary degrees from various universities, and was a member of many foreign scientific bodies. He was the first Longstaff Medallist of the Chemical Society, and for his researches on fluorine compounds and his determination of the atomic weights of titanium and gold he received the Royal Medal of the Royal Society, of which body he acted as foreign secretary from 1899–1903.

The Bakerian lecture delivered by him before the Royal Society dealt with radium, the atomic weight of which he determined, and he was joint author of two other Bakerian lectures. He was made a Companion of the Bath in 1900, and was knighted in 1909.

Thorpe was abundant in literary labours. His delightful studies in historical chemistry will be prized for many generations, as for example the monographs on Priestley and Davy, the History of Chemistry, and the life of Roscoe. A review which he wrote for this Journal rather less than three years ago shows how wide and accurate was his knowledge of the historical progress of chemistry. But he will be best remembered in this connection by his work in producing the Dictionary of Applied Chemistry, a book which has been found indispensable to all practising chemistry. To this he gave himself with characteristic vigour, especially during his retirement, and the proofs of the last volume of the edition now being issued were in his hands during the last few weeks of his life.

The contributions of Thorpe, either directly or indirectly, to analytical chemistry, the branch of the science with which this Journal is specially concerned, were considerable. In his early days as a Professor he had published two little handbooks on Qualitative and Quantitative Chemical Analysis; and throughout the period of his career at the Government Laboratory he revealed to those who had the privilege of working with him a keen interest in and an accurate knowledge of analytical method.

The design of the Government Laboratory at Clement's Inn will remain one of Thorpe's great achievements. Originally housed in attics and improvised rooms at Somerset House, where it had been established in 1842 to deal with a particular revenue question, it had outgrown its bounds by reason of the demands made upon it by many Government Departments, and Thorpe had the good fortune to transfer the laboratory in 1897 to a building which he had designed in every detail. The year after Thorpe had gone, his late Department, which he had done so much to establish as a necessary Crown activity, became the Department of the Government Chemist with Treasury direction to undertake chemical work for all other services of the Crown.

Thorpe was a member of the Royal Commission appointed in 1900 to enquire into the causes of the outbreak of arsenical poisoning, and acted as Chairman of a Committee of Chemists appointed by the Board of Inland Revenue to report upon the methods employed in the detection of small quantities of arsenic in materials used in brewing. Under his direction, the old Marsh-Berzelius method with zinc and acid as a source of hydrogen was much improved, and an electrolytic method was devised. Bloxam of King's College, in 1861, had described such a process, but it was not capable of general adoption, and no advance had been made with it. Thorpe's apparatus, except for slight modifications, is used to-day essentially in the form in which it was originally described by him.

Thorpe's position at the Government Laboratory, which since 1875 had been the authority for reference by the Courts in cases of disputed analyses under the Food and Drugs Acts, made it necessary for him to enquire closely into the methods of analysis employed. One of the matters which came up for review in this connection was the analysis of sour or decomposed milk, a subject of very great importance to public analysts whose figures might be challenged in the Courts. As a result of an investigation into the method, and of further extended experiments carried out, Thorpe communicated a paper to the Chemical Society in 1905, in which he described the methods adopted to obtain the figures for the original fresh milk from the analysis of the decomposed milk. This authoritative statement was welcomed by public analysts who found that Thorpe was always ready to discuss with them analytical processes.

A similar proceeding arose in connection with the Food and Drugs Act of 1899, which fixed a limit for the amount of butter fat in margarine. It was at Thorpe's suggestion that the Society of Public Analysts, of which he was an honorary member, appointed a committee to confer with him as to a suitable method for the estimation of the amount of butter fat. Thorpe had held for a short time an appointment as public analyst, and this experience no doubt became a point of contact with the work of this Society.

Among the important revenue questions which came before Thorpe, were two of special importance to analysts. One of them related to the determination of the original gravity of beers; in this work he collaborated with the late Dr. Horace Brown, F.R.S., and as a result of their report, the original tables based upon the work of Graham, Hofmann and Redwood sixty years before, were modified, and tables giving the new values were incorporated in the Finance Act of 1914. The other investigation related to the estimation of alcohol. Although it was the practice of analysts to determine the amount of alcohol with a pyknometer, using, in connection therewith, alcoholic tables of strength, the legal instrument for revenue purposes in this country had been, since 1816, Sikes' hydrometer. In order to make available the direct specific gravity method, Thorpe prepared for use a table of spirit densities based upon the work of Blagden and Gilpin, Drinkwater and Mendeléeff, at the same time revising the tables for use with Sikes' hydrometer. By an order in Council made under the powers of the Finance Act, 1907, it is now legal, for revenue purposes, to determine the strength of spirits by specific gravity, using in connection therewith these tables.

Thorpe was a member of numerous Departmental Committees, and his keen and energetic interest in the questions investigated by the Committees on "Preservatives and Colouring Matters in Food," on "Milk and Cream Regulations," on "Butter Regulations," and on "Industrial Alcohol," will be remembered by those who were associated with these enquiries. He was also Chief Analyst under the Fertilisers and Feeding Stuffs Act, and was chairman of the Committee which drew up for the Ministry of Agriculture the methods of analysis used under that Act.

One other of Thorpe's many activities may be mentioned. He was deeply interested in the use of lead in pottery glazes, and it was largely due to the investigations carried out under his directions, and to the evidence which he was able to give at the enquiry held into the matter, that steps were taken to limit the quantity of lead in soluble form in fritts and glazes.

This brief review leaves untouched a great deal of work in which Thorpe was actively engaged, although mention should be made of his work in connection with the International Committee on Atomic Weights, on which he served for many years. His great powers for work, his descriptive pen, his attractiveness as a lecturer, are well known. An illness which attacked him at Edinburgh on the eve of delivery of his Presidential address, in 1921, left its mark, but he carried on strenuously almost to the absolute close of his long life, talking as each day ended of the work with which the next day would be occupied.

He died in his own home at Salcombe—the port into which he had often sailed his yacht from quests in British and Continental waters—and thus belied the feeling which he once expressed that he would die on the sea he loved so much. The simple ceremony at the funeral at Salcombe, which the writer attended as representing the Government Chemist's Department and the Chemical Society, was in keeping with the man who disliked ceremony and who cared for life only for what could be accomplished in it.

GEO. STUBBS.

The Investigation of Ventilation Conditions.

BY ROBERT C. FREDERICK.

(*Read at the Meeting, March* 4, 1925.)

THE factors relating to ventilation have been the subject of a vast amount of research, and it is only in recent years that the conditions and requirements of satisfactory ventilation have been fully understood. Before discussing the modern aspect it may be of interest to give some account of the views which have had currency.

From early times it has been recognised that a certain amount of air supply is essential for the maintenance of human life, and tragedy has followed swiftly when this fact has been forgotten or ignored. The infamous case of the Black Hole of Calcutta is common knowledge. There, in 1756, 146 people were confined in a building 18 feet square and with only two small windows to provide ventilation. Within an hour some were dead, and a little over nine hours later, when the prisoners were released, only 23 half-dead individuals staggered forth. In 1848 the *S.S. Londonderry*, on passage from Sligo to Liverpool, encountered heavy weather. The 150 steerage passengers were battened down in a cabin 18 feet long, 11 feet wide, and 7 feet high, and before an exit could be forced 72 were dead and others were dying. Again, after the battle of Solferino (1859) 300 men were confined in a small prison, and next day 260 lay dead.

CHEMICAL EXPLANATIONS.—Even a generation or two ago it was considered that air itself could contain a chemical causative agent of many diseases. Griscom,[1] writing in 1854, said: "It would appear that sulphuret of ammonia is the morbific agent exciting typhus fever, sulphuretted hydrogen being the pestilential virus producing yellow fever and the bilious remittents and agues of tropical climates." This ague, which we now know as malaria (Ital. *mala*, bad; *aria*, air), was considered to be due to a noxious vapour or miasma which was evolved when vegetable matter was in contact with water and the temperature was sufficiently high; this reaction was more pronounced at night.[2] Through the advance of scientific knowledge it is now known that malaria is caused by a parasite, part of the life cycle of which is spent in the anopheline mosquito, whose bite may convey the infection to man. The mosquito breeds in marshes and swamps and is active only at night. It is not surprising, in view of these beliefs, that there was a general distrust of the "night air," and that windows and doors were commonly kept tightly closed after dark, with consequent lack of ventilation, though, no doubt, these precautions were not unrelated to the question of the security of the household from depredators. Yet the harmful effect of living in badly ventilated spaces was realised, though the explanation of this was founded on erroneous conceptions. A commission, which enquired into the relationship between disease and ventilation of the barracks in the Crimea, showed there was a much higher incidence and mortality among the men living in badly-ventilated quarters than among those in well ventilated ones.[3]

EFFECT OF RESPIRATION ON THE COMPOSITION OF AIR.—A man, at rest, breathes about 17 times a minute, and at each inspiration inhales some 500 c.c. of air. In this process the composition of the air is markedly altered, and the expired air contains, by volume, about 16·4 per cent. of oxygen instead of the original 20·9, and the carbon dioxide is increased from 0·03 to about 4·0 per cent.; the quantity of nitrogen and inert gases undergoes little change, but a large amount of water vapour is added. The composition of the air in a single expiration is subject to some variation. The alveolar air contains about 6·0 per cent. of carbon dioxide and correspondingly less oxygen; consequently the composition of the expired air is influenced by the depth of the expiration.

VARIATIONS OF HUMIDITY.—The amount of water vapour added to the air of a compartment by the occupants varies greatly according to the temperature, the work performed, and other factors. The degree of vitiation caused by this is much more than is commonly believed. For example, the relative humidity at 65° F. in an air-tight glass cabinet of 343 cb. ft. capacity, occupied only by the author, increased from 31 to 49 per cent. in half an hour. It has been stated[4] that under the usual atmospheric conditions of temperature and humidity the amount of water given out, in respiration and sweat, by a man, at rest, in 24 hours, is 935 grms., an average of 39 grms. per hour. With three men at rest occupying the cabinet referred to above, in which the average temperature was 69° F., and the average relative humidity 62 per cent., it was found that 1300 grms. were added to the air in 2 hours, an average of 216·7 grms. of water per man per hour. The latter quantity is sufficient to cause saturation of 54·7 cubic metres (1932 cb. ft.) at 60° F. and 70 per cent. original relative humidity. In practice, however, it does not work out in this way. With twenty-one other men the author has occupied a steel compartment of about 8000 cb. ft. capacity for 12 hours. The initial and final temperatures were 46° F., and 52° F. respectively; the relative humidity actually fell from 82 to 79 per cent. Condensation of water vapour on the walls was marked, though the amount was insufficient to account for that added by the occupants. Again, in an air-tight glass chamber containing 337 cb. ft. of air at an average temperature of 64° F., and occupied by three men for 4 hours, the absolute humidity only increased 3·5 grms. per cubic metre, and no condensation could be detected. The disappearance of water vapour sufficient in quantity to saturate the air of a space several times over will be the subject of further research; it is probable that a large amount is taken up by the clothing.

SIGNIFICANCE OF CARBON DIOXIDE.—The harmful effects of living in air vitiated by the products of respiration were formerly considered to be due to a poisonous action of the added carbon dioxide, the deficiency of oxygen, and the presence of some unidentified poison.

In the most overcrowded living spaces the carbon dioxide present as a result of respiratory addition never exceeds 0·9 per cent., and carbon dioxide even very much in excess of this figure is not poisonous and has no physiological significance. This is shown by the relatively enormous amounts breathed by people huddled in bed clothes and by workers in breweries and aerated water factories. Again, at each expiration in man there remains some 150 c.c. of air in the respiratory passages, and this with its high carbon dioxide content is the first to reach the alveoli in the following inspiration. In breathing air containing 0·5 per cent. of carbon dioxide the ventilation of the lungs is increased to such a small extent as to be quite unnoticeable.[5] A concentration of 2·0 per cent. of carbon dioxide simply results in a 50 per cent. increase in the depth of breathing, such as occurs with moderate exercise.[6] Living in such a concentration does not cause discomfort unless vigorous exercise is performed, and it is not until the amount increases to between 5 and 7 per cent. that breathing becomes laboured when the subject is at rest.[7] Pettenkofer alone, among his contemporaries, realised that the carbon dioxide content was not the cause of the discomfort experienced when living in badly ventilated spaces.[8]

In calculating the amount of carbon dioxide added to the air of an apartment it is generally assumed that the output of an adult male at rest is 0·72 cb. ft. per hour, and that of a mixed community of men, women and children 0·6 cb. ft., women exhaling less than men, and children still less. I consider these figures too high. In a number of experiments it was found that the mean output per man at rest was 0·57 cb. ft. per hour. Macleod[9] gives the output per man at rest as 0·327 grm. of carbon dioxide per hour per kilo. of body weight; calculating on an average weight of 68 kilos, this would give 0·4 cb. ft. per man per hour. When work is being performed there is a great increase in the amount of carbon dioxide exhaled. The ratio of carbon dioxide increase to oxygen decrease caused by respiration in a space occuped by average men I find to be 1:1·15.

De Chaumont, in 1875, stated that when the normal amount of carbon dioxide in fresh air, which he considered to be 0·04 per cent., was increased by more than 0·02 per cent. from respiration this excess was perceptible to the senses by an associated disagreeable smell. It is remarkable how this statement received, and still receives, such general acceptance. The error was perpetuated in the fixation of the so-called sanitary limit for carbon dioxide, 0·04+0·02, a total of 0·06 per cent., a figure which to this day appears to be regarded as standard by many analysts and sanitarians. Twenty-one years later this belief was elaborated by Russell[10] in this curious statement:—"Experiment by means of the sense of smell has shown that air in a room seems fresh when the carbon dioxide does not exceed 0·05999 per cent., a little unpleasant when the proportion is 0·08004 per cent., offensive and very close at 0·12335, and extremely close, when the smell can no longer differentiate, at 0·12818." It is difficult to know which to admire most, the delicacy of the investigator's olfactory sense or that of his analytical technique. In point of fact, the amount of carbon dioxide in fresh air averages 0·03 per cent., which would make the sanitary limit 0·05 per cent. Taking the permissible respiratory impurity as 0·02 per cent., and the carbon dioxide output as 0·6 cb. ft. per person per hour, De Chaumont calculated that 3000 cb. ft. per head per hour of fresh air must be supplied to prevent the sanitary limit being exceeded. Pettenkofer, whose perspicacity has already been noted, suggested, in 1858, a permissible maximum of 0·1 per cent. of total carbon dioxide.

The author has, in many years of ventilation investigation, worked to a permissible maximum of 0·12 per cent. of total carbon dioxide. It must be clearly understood that this figure has no chemical or physiological significance whatever, and that it is adopted for reasons different totally from those animating the school of de Chaumont; it functions simply as an index. A large proportion of disease dissemination is caused by air as the vehicle. The organisms are carried either by currents or by direct transfer in sputum or nasal spray. The probability of infection being contracted from a carrier by the other occupants of a space depends, among a number of other factors, upon whether the natural defences of the individual are sufficient to overcome the mass of infective material encountered; the larger the number of organisms which gain access to an individual the greater is the probability of their survival in a fresh host. It follows that the greater the degree of dilution of the air with the pure outside air, the less the probability of illness being communicated. This, then, is the function of the permissible limit of 0·12 per cent. of carbon dioxide; it ensures that the respiratory addition to the air space will be diluted to a certain minimum extent, and consequently that the risk of the spread of any infectious disease organisms present will be proportionately reduced. The figure is one which does not entail any unreasonable demands on ventilation practice.

DEFICIENCY OF OXYGEN AS EXPLANATION.—By the lay mind all the discomforts of inadequate ventilation are attributed to lack of oxygen. Actually the deficiency of oxygen under these circumstances is never more than 1·0 per cent., and this is without any effect whatever. The normal variation in the barometric pressure at sea-level is equivalent to a 10 per cent. difference in the partial pressure of oxygen in the atmosphere. Leonard Hill[5] states that a person not exerting himself will fail to notice any effect until the oxygen has been reduced to about 11 per cent., and consciousness will not be lost until the percentage sinks below 7 per cent. The author has lived in an atmosphere containing 15·21 per cent. of oxygen and experienced no discomfort.

The belief in the existence of a poison in exhaled air was founded on experiments in which the condensed water vapour from human expirations was injected into animals with fatal results. The question has since been the subject of much research, and the net result goes to show that there is no evidence that such a poison exists.

It is necessary to point out that while the carbon dioxide increment and the oxygen decrement which result from respiratory vitiation of air are of little importance, massive changes which certainly have a profound effect can occur through other causes. The author has shown[11] that storage of vegetable substances in an unventilated space may convert the air into a mixture of gases containing some 50 per cent. of carbon dioxide and hardly any oxygen, and that anyone entering such a space would be immediately killed. The research was made in order to elucidate the reason of a triple fatality, and the causative agent in this case was damp tobacco; hay, cabbage, potatoes, etc., react similarly. In a notice of this paper[12] the reviewer pointed out that there was a tradition amongst seafaring men of sailing ships that coffee beans wet with salt water in the holds cause sudden deaths amongst the crew. Douglas[13] has reported a case during the war where three men lost their lives in clearing a cargo of grain in a ship which had been sunk and afterwards salved. In 1915 a cargo of oranges on another ship was responsible for the death of two men entering the hold. This, and another similar fatal accident in 1914, in which four men were killed on entering a silo containing immature Indian corn, are described by Brooke.[14]

For a particular purpose in carrying out some experiments the author had the floor of an air-tight cabinet painted over with boiled linseed oil and noticed that this caused a higher carbon dioxide content in the air. Subsequent research showed that exposure of the oil had this effect and, in addition, removed a large proportion of oxygen. A volume of oil in contact with 793 times its volume of air added 0·33 per cent. of carbon dioxide and reduced the oxygen to 11·72 per cent. in a week, and there was evidence that this change had been brought about some considerable time before the samples were actually analysed. It has been suggested that red lead paint has a high absorption capacity for oxygen, but red lead *per se* has no such effect; the reaction is due to the admixture of the oil constituent.

In a sample of air from a paint store the author found 2·05 per cent. of carbon dioxide and 15·86 per cent. of oxygen. The importance of recently painted surfaces in investigating ventilation conditions does not require elaboration. Large amounts of carbon dioxide in the air of breweries, aerated water factories, and bakehouses are to be expected. A fatality due to anoxæmia caused by the employment of a rusting composition in the hollow pier of a bridge is discussed by Wallace.[15] The changes in the carbon dioxide and oxygen content of air brought about by combustion processes are an obvious factor to be considered. Except in the case of naked gas flames and tobacco smoking, it may be stated that, in general, the products of combustion do not enter a space, but find an exit in the connecting flues; a large volume of air is consumed, and its replacement from outside sources exerts a powerful beneficial ventilating influence. A naked gas flame can increase the carbon dioxide content of the air in a space very greatly; this carbon dioxide, however, is without the significance of that from respiration. A much more important factor is the heat evolved; one cb. ft. of gas is said to raise the temperature of 31,290 cubic feet of air 1° F.[16]. In this paper it will not be possible to discuss the question of poisonous gases occurring in air.

EFFECTS OF TEMPERATURE AND HUMIDITY.—Hermans[17] was the first to state definitely that the most important considerations in ventilation are not carbon dioxide, oxygen, or personal effluvia, but temperature and humidity. His teaching, for the most part, fell on deaf ears, and it was not until 1905, with the classic experiments of Flügge, Heymann, Paul, and Ercklentz,[18] that his theories obtained general acceptance. In these experiments individuals experienced no discomfort and suffered no impairment of mental efficiency when confined in atmospheres containing 1·0 to 1·5 per cent. of carbon dioxide, and correspondingly less oxygen, provided the temperature and humidity were kept low. In other experiments, with carbon dioxide and oxygen in about the same amount as before, and with the temperature from 75 to 80° F., and relative humidity 88 per cent., discomfort was marked even when the subject, through a suitable attachment, breathed pure air from the outside. Their views were supported in reports to the Government in 1907 and 1909.[19 20] In 1913 our knowledge was further extended with the publication of a monograph by Hill, Flack, McIntosh, Rowlands, and Walker.[21]

The importance of temperature and humidity is due to their influence on the body metabolism. The food consumed by the individual is continuously being converted into heat, and but for the natural heat-regulating mechanism, the body temperature, normally 98·4° F., would rise. The profound effect of even a degree rise in body temperature is common knowledge. To maintain a balance there must be a continuous process of heat loss. In cool temperatures this occurs principally by convection and, to a much less extent, by radiation. The cool air conveys away the body heat from the skin by convection, and loss by evaporation from the skin is at a minimum; from the respiratory membrane loss by evaporation is at a maximum by the breathing of cool air of low vapour tension.[22] When the air of a space attains a temperature above 70° F. these processes are inadequate, and the body temperature would rise but for the secretion of perspiration which then occurs. As long as perspiration can evaporate freely the heat loss and production are maintained in equilibrium[23].

It follows, then, that when a person is living in a space in which the temperature and humidity are high, heat loss is retarded and, in consequence, discomfort is experienced; in extreme cases heat-stroke and death may supervene. The most essential requirement of satisfactory ventilation is, therefore, that conditions shall be maintained such as to permit of free heat loss from the individual. The heat-regulating mechanism is greatly assisted by any means which disperse the envelope of hot humid air surrounding the subject between the skin and the clothes. Movement of the air in a space by direct transfer and promotion of evaporation is the most potent factor in rendering this assistance, and its importance in ventilation cannot be over-emphasised. It has fallen to the author's lot, in the course of his investigations, to work in atmospheres foully vitiated by respiration, vitiated, indeed, to such an extent that dilution of the respiratory impurity was absolutely *nil*; it is only under extreme conditions that the truly remarkable effect and relief afforded by pronounced air movement can be fully appreciated.

Satisfactory ventilation, therefore, demands that a sufficient quantity of pure air be supplied to dilute the respiratory impurity, that the air in the space be kept at a hygienic level of temperature and humidity, have a high cooling power, and be kept in appreciable movement. These conditions must be maintained without causing annoyance to the occupants.

Before discussing the methods to be adopted in investigating to what extent these requirements are being fulfilled it may be desirable to make some reference to what may be termed the psychological aspect. I am convinced that if the occupants of an apartment *think* the ventilation conditions are unsatisfactory, they will imagine they are exhibiting the symptoms these conditions bring in their train. As a result of persistent complaint by the operators, the author was requested to report on the ventilation conditions in the X-ray room of a large hospital. When the apparatus was in use the room was in total darkness except when momentarily illuminated by electric discharge flashes, and there was a pronounced and rather unpleasant smell of ozone. Under these circumstances it is not difficult to understand why the occupants thought the air *must* be very unhealthy. The amount of ozone was negligible no noxious fumes were present, and the ventilation, in general, was quite satisfactory. After the matter had been explained complaints ceased.

EXAMINATION OF AIR.—From the foregoing discussion it will be seen that in investigating the ventilation conditions of a space it is necessary to make the following examination of the air:—1. Determination of carbon dioxide; 2. Determination of temperature and humidity; 3. Determination of cooling power.

Of these, the first is the most important, for, if the figure is low, it is probable, speaking generally, that the remaining determinations will also give satisfactory results. But they are really complementary, and should all be regarded as essential. Different parts of the space should be examined at about body level. A complete investigation can be made in a short time.

1. DETERMINATION OF CARBON DIOXIDE.—For carrying out this determination a method is available which is so advantageous from every point of view that no other need be considered. I refer to the Haldane Apparatus for Determination of Carbon Dioxide in Air. It is necessary to distinguish it clearly, as the admirable principles on which its use depends have been utilised by Haldane in the design of various other air and gas analysis apparatus. The apparatus is contained in a case measuring only about 8 by 13 by 3½ inches and can be employed direct in the space under investigation or for examination of samples in the laboratory. The most convenient sample bottles have a capacity of 70 c.c., and are narrow-mouthed, and stoppered; they must be perfectly clean and dry. It is advisable, as soon as a new supply is obtained, to etch a distinguishing number on the stopper and bottle so that the correct stopper is always employed in each bottle. To ensure absolute tightness, the stopper

is smeared with a thin coat of vaseline. To prevent the stopper working loose, particularly when exposed to changes in temperature and pressure, it is essential to secure it by placing a rubber band over it and under the bottle; another smaller band, doubled over this and round the neck, maintains the first in position. In collecting a sample, one end of a rubber tube (about $2\frac{1}{2}$ ft. long and $\frac{8}{16}$ in. bore) is introduced into the bottle nearly to the bottom, and the other end is held in the mouth. A deep breath of air is then sucked through and the tube removed while the air is still being sucked up. The importance of not allowing any breath to pass backwards into the bottle and of keeping the bottle at arm's length need hardly be emphasised. The stopper is inserted, turned round, and secured with the rubber bands. The author has, elsewhere[24], described in detail the manipulation of the apparatus when used either direct in the space or with sample bottles. Since writing that account he has personally analysed some 5000 additional samples, but the directions given do not call for any important revision. One or two points, however, may be referred to with advantage. It is desirable to test, from time to time, whether the apparatus is air-tight; the best way of doing this is to note the reading after passing the air, which happens to be in the apparatus, backwards and forwards in six absorptions, then to repeat this with the residue, and see if the reading remains constant. If the apparatus is not quite tight, the trouble is usually due to the lubrication of the stopcocks. Resin cerate is the best lubricant, and a supply should be prepared by melting a quantity and allowing any hard impurity to settle; only the top portion of the solidified product is used. Too much attention cannot be given to the stopcocks to ensure freedom from gritty particles, pieces of fibre, etc. When using the apparatus direct in a space in which the temperature is high, inconveniently extensive movement of the potash levels may occur, but this can be avoided by shaking the water in the jacket immediately before closing the stopcocks after taking the sample into the apparatus. As has already been stated, the quantity of carbon dioxide should not exceed 0·12 per cent., and, of course, the less it is the better.

If, for any exceptional reason, a determination of the oxygen is also desirable, the Haldane General Air Analysis Apparatus should be employed. Full details for its manipulation have been given by the author elsewhere.[25] This apparatus may also be used for the determination of all combustible gases, and in this case the modified combustion chamber described by the author[26] will be found an advantage.

2. DETERMINATION OF TEMPERATURE AND HUMIDITY.—This is best carried out by means of a wet and dry bulb hygrometer of the Mason type. The common method of simply hanging up such an instrument on the wall of a space is useless; it is absolutely essential that the air surrounding the instrument should be in distinct movement. The test is conveniently made with a whirling hygrometer. The type recently introduced by Messrs. Casella has been found very satisfactory. In this, wet and dry bulb thermometers are mounted in an instrument very similar to the noise-producing device employed by certain football enthusiasts, and the method of using is also the same. The wet bulb is kept moist by an

absorbent fibre-covering dipping into distilled water contained in an attached reservoir. The thermometers are whirled round, and readings are quickly taken until constant. For investigations extending over a prolonged period, the recording thermo-hygrograph made by the same firm, the mechanism of which is actuated by a bi-metallic strip and the contraction and elongation of a bundle of human hair, will be found useful. It is necessary to standardise it from time to time with a whirling hygrometer. From the wet and dry bulb temperatures it is possible, by means of three tables, to calculate the Dew Point, the Absolute Humidity and the Relative Humidity (*i.e.* the percentage proportion of water, as vapour, in the air to that present when the air is saturated at the dry bulb temperature). The original tables were calculated by Glaisher many years ago, and the absolute humidity is given in grains per cb. ft. To conform to more modern usage, the author has calculated a condensed table to grms. per cubic metre, and with this has given the other tables necessary, and has described the method of calculation.[25] The normal variations in atmospheric pressure at sea-level in this country may be neglected in humidity calculations without serious error, but for greater differences Marvin's tables[27] should be consulted. In all cases where the relative humidity alone is to be determined, it may be obtained without calculation from the tables already mentioned. In the author's opinion the most satisfactory condition is when the dry bulb temperature is 64° F. and the wet bulb 57° F. This indicates a dew point of 51·2° F., and a relative humidity of 63·1 per cent. The relative humidity is a much more important consideration than the absolute humidity, for it is on the former that the power of the air for evaporating perspiration depends. Rubner found that at 73·4° F. a man at rest lost 75 grms. of water per hour when the relative humidity was 60 per cent., and only 19 grms. per hour when the relative humidity was 84 per cent. The wet bulb temperature in temperate climates should not exceed 70° F. There is a considerable margin between what is desirable and what can actually be endured without causing a rise in body temperature. Haldane states[28] that with the subject at rest and lightly clad the maximum tolerable wet bulb is 88° F. in still air, and 93° F. when the air is in movement; when work is being done these figures are 78° F. and 85° F. respectively. It would appear, however, as a result of recent research that light work can be done when the wet bulb registers 90° F., provided the air is in appreciable movement. So far consideration has only been given to the question of damp air, but air can also be too dry. The Chicago Commission on Ventilation, speaking of the very dry air supplied to the schools of that city, stated that the air tended to maintain a proper balance by absorbing moisture from the school structure and the bodies of the children and that this resulted in shrunken furniture, dust, dry throats, parched lips and a rapid rate of skin evaporation which rendered it necessary to maintain a high temperature for comfort. The author has found that with a relative humidity of 30 per cent. and the air in distinct movement a comparatively high temperature, say 75° F., gave an impression of being some 15 to 20 degrees cooler.

3. DETERMINATION OF COOLING POWER.—This determination is made by means of an instrument called by its inventor, Professor Leonard Hill, a Katathermometer.[29] In its latest form this consists of an alcohol thermometer with a cylindrical bulb about 1·7 cm. in diameter and 3·6 cm. long and a stem about 18 cm. in length with two graduations at 100 and 95° F. respectively. At the other end of the stem is a safety reservoir larger than those of ordinary thermometers. The purpose of this is to enable the thermometer to be heated well above the highest graduation without risk of breakage, and also to allow time for placing it in position and to attain a regular rate of cooling before the reading requires to be recorded. Both wet and dry Katathermometer readings are taken. To obtain the dry reading the instrument is placed in distilled water at about 170° F. (in working away from the laboratory a Dewar flask will be found convenient) until the liquid about half fills the reservoir and presents an unbroken column. It is then wiped quite dry and clamped in a burette stand well away from the body. It should be placed out of direct air currents, unless the occupants of the space are usually similarly situated, as the rate of cooling depends very greatly upon air movement around the instrument; with the tables provided the instrument can, indeed, be used to measure movements of the air much smaller than could be recorded by an air meter. The exact time taken by the liquid to recede from the 100 to the 95 mark is recorded in seconds by means of a stop-watch. This figure, divided into the particular factor engraved on each Kata-thermometer, gives the cooling power in millicalories per sq. cm. per second of a surface at 36·5° C. (97·7° F.). This figure is arbitrarily chosen as representing the skin temperature. The first reading should be ignored, and subsequently the mean of a number should be noted. To take the wet Kata-thermometer reading, the bulb is covered with a Lisle-thread finger stall and, after heating, the excess water is removed by drawing the covering up tightly and shaking the instrument when held in a vertical position. The remaining manipulation is exactly the same as for a dry reading. The dry Kata-thermometer gives the cooling power by radiation and convection, the wet by radiation, convection, and evaporation, and consequently the difference between the two is the cooling power by evaporation. Professor Leonard Hill states that the cooling power of air by the dry Katathermometer should not be less than 6, and that of the wet 18. In the author's opinion, however, these figures are perhaps idealistic, and 5 and 15, respectively, should be regarded as sufficiently satisfactory.

Air movement can be most conveniently studied by observing the behaviour of a column of smoke. In investigating ventilation conditions it is inadvisable, for obvious reasons, to add smoke produced by combustion to the air of the space under examination; the author has therefore designed an apparatus which, by simple pressure of a blowing bulb, delivers a column of artificial smoke, It is obtainable from Messrs. Casella & Co., Ltd.

ROYAL NAVAL MEDICAL SCHOOL,
ROYAL NAVAL COLLEGE, GREENWICH.

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DISCUSSION.

Mr. W. J. A. **Butterfield** thought the resumé of the subject given in this paper extremely valuable. He believed, however, the author was not right in stating that the carbon dioxide in air was regarded up to 1908 as directly injurious, and not merely as an indicator of the degree of ventilation. For instance, in evidence given on 29th May, 1902, before the Select Committee on House of Commons Ventilation, Dr. J. S. Haldane stated (Question 820):—"The carbonic acid by itself is, of course, of no importance," and (Question 823), "but of course the carbon dioxide there is only treated as an index." These words suggest that earlier references could be traced to a fact which Dr. Haldane then spoke of as more or less common knowledge among ventilation experts.

In a paper read before the Society in 1909 (Analyst, **34**, 257) the speaker quoted experience with 9000 samples of air taken by the bottle method referred to by the author as evidence of the satisfactoriness of that method of sampling, and his later experience with further samples had confirmed this evidence.

A form of hygrometer not mentioned by the author, which was much more convenient and less costly than the Assmann instrument, was that devised by C. V. Boys, formerly prescribed for use in connection with calorimetry in the official gas-testing stations in London.

Mr. R. C. **Frederick**, in replying, said how much he appreciated the generous reception which had been accorded to his paper. He had not seen the hygrometer designed by Professor Boys, and so could not form an opinion as to its suitability for investigation of ventilation conditions. He (the speaker) had recently designed one of a new type, but had not proceeded with it at the moment, as he found it would be relatively costly to manufacture. Mr. Butterfield had suggested that the conditions and requirements of satisfactory ventilation had been understood at an earlier date than that indicated in the paper, but he thought that enquiry would show that the facts were as stated.

The Carbon Error in the Quantitative Deposition of Nickel and Iron from Complex Oxalate Electrolytes.

By P. K. FRÖLICH.

(*Read at the Meeting, March* 4, 1925.)

Nickel and iron, when deposited electrolytically from the complex oxalate electrolytes used in the quantitative determination of these metals, appear to be contaminated with organic matter, causing a yield greater than the theoretical.[1,2,3,4,5] As the various investigators come to different conclusions regarding the cause and magnitude of the error thus introduced* (the so-called "carbon error"), it was thought advisable to undertake a systematic study of the problem.

I. DEPOSITION OF NICKEL.

Most experimenters agree that the organic contamination of nickel deposited from an oxalate electrolyte is too great for quantitative determination of the metal.[2,6] Hence, in the separation of nickel from aluminium nickel and chromium, where an oxalate electrolyte is used, the analysis becomes complicated because it is necessary to re-dissolve and re-precipitate the nickel from a complex ammoniacal electrolyte in order to free the metal from the organic matter.[6] To see if it might be possible to reduce the carbon error so as to warrant the direct determination of nickel from an oxalate electrolyte, the effect of different variables was studied. The temperature, anodic current density, cathodic current density, and length of time of electrolysis were varied. The results of these experiments are given in Table I.

Experimental.—Nickel sulphate of the highest purity was dissolved in distilled water to give a concentration of about 10 grms. of metal per litre of solution. Samples of 25 c.c. were pipetted out and made up to a volume of 250 c.c. with 10 grms. ammonium oxalate. The cathode consisted of platinum foil, 0·35 sq. dm.

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* The literature on the subject is reviewed by the writer in a paper presented at the American Electrochemical Society's Detroit Meeting, October, 1924. (T.A.E.S., 46, 181, 1924).

in area. Platinum wire or foil was used as anode. The electrolyte was tested for exhaustion with dimethyl glyoxime. Unless otherwise stated, the deposition was quantitative. The excess weight of the deposit, given in the last column of the table, is calculated as per cent. of the actual metal content of the precipitated matter, which was determined by electro-deposition of the metal from a complex ammoniacal electrolyte (standard method), the following being the result of three analyses:—0·2463 grm.; 0·2464; 0·2462. Av. 0·2463 grm. of nickel.

TABLE I.

ELECTRO DEPOSITION OF NICKEL FROM COMPLEX OXALATE ELECTROLYTES.

Containing 0·2463 grm. Metal.

No.	Addition.	Cathodic C.D. amps./sq.dm.	Anodic C.D. amps./sq.dm.	Temp. °C.	Time Hours.	Deposited Grms.	Per cent. Excess Weight.	Notes.
35a		2·0	25	25	13	0·2485	0·90	Deposition not quantitative
35b		2·0	25	25	13	0·2490	1·10	
45		1·0	25	25	18	0·2498	1·42	
50		1·0	25	25	23½	0·2505	1·71	
57a		1·0	2·8	25	17	0·2489	1·06	
57b	10 c.c. 3% H_2O_2	1·0	2·8	25	17	0·2503	1·62	
80	10 grm. NH_4Cl	1·0	2·8	25	21	0·2535	2·92	
36b		1·0	2·8	85–87	7	0·2478	0·61	
43b		1·0	2·8	85–87	7	0·2485	0·90	
54		1·0	2·8	85–87	8	0·2515	2·11	
36a		3·0	8·0	85–87	4	0·2521	2·35	
43a		3·0	48–50	85–87	4	0·2512	2·00	

In order to avoid a lengthy discussion of details, it may be sufficient to refer to the table from which the wide variations in experimental conditions will be apparent. In no case is the excess weight of the deposit appreciably below one per cent., averaging about one and a half to two per cent. Although the length of time needed for quantitative deposition might be somewhat reduced by using ordinary analytical wire-gauze electrodes, instead of platinum foils, this would not, according to the writer's experience, change the degree of contamination to any considerable extent. Hence, the results show that under the conditions studied, it is not possible to bring the carbon error within the limits of the permissible analytical error.

Special mention may be made of the experiment (No. 57b), where hydrogen peroxide was added, in view of the beneficial results obtained by Madsen when applying this addition agent to commercial electrolytes.[7] While in the latter case the peroxide is said to check the reactions leading to organic contamination of the deposit, it was found here that this agent worked in the opposite direction, *i.e.* an increase in excess weight of the deposit was observed as compared with a parallel blank run (No. 57a).

A diaphragm (porous cup) surrounding the anode did not prevent contamination of the deposit, as stated by Lambris.[8] Furthermore, quantitative deposition was not obtained in this case as the nickel was precipitated as carbonate in the anode compartment.

The highest percentage of organic matter in the cathodic deposit was observed in the experiment (No. 80) where ammonium chloride had been added to the electrolyte. This result is interesting in comparison with the depressing effect exerted by ammonium chloride on the reactions leading to contamination of iron deposits (see the following section).

II. DEPOSITION OF IRON.

The method of electro-deposition from a complex oxalate electrolyte is far more important in the analysis of iron than it is in the case of nickel, because this is the only method known of quantitative determination of iron by electro-deposition while nickel may be deposited in an absolutely pure condition from a complex ammoniacal electrolyte. Fortunately the carbon error in the deposition of iron is less serious than in nickel deposition, though much controversy has arisen as to the real magnitude of the error introduced in the electro-analysis of this metal. No definite data on the subject are available in the literature. In view of an observation by Verver,[5] however, most textbook authors recommend that the electrolysis be discontinued as soon as quantitative deposition has taken place, as prolonged electrolysis causes an increased accumulation of organic matter in the cathodically deposited iron.

EXPERIMENTAL.—Table II. gives the results of a series of experiments made under varying conditions, the details of experimental procedure being the same as before, except that 8 grms. of ammonium oxalate (instead of 10 grms) were added to each sample of the ferrous sulphate solution. The temperature was kept constant at 25° C. The true iron content of the stock solution used was 0·1947 grms. per 25 c.c. as determined by titration with permanganate. The character of the residue left upon dissolution of the iron in cuprammonium and ferric sulphate solutions is described in the last column of the table.

The results may be summarised as follows:

(1) The degree of organic contamination

(a) Is constant in parallel runs (Nos. 77 and 81).

(*b*) Increases with the time of deposition (Nos. 79, 77, 81, 67).

(*c*) Increases with increasing anodic current density (Nos. 75 and 77).

(*d*) Increases with an addition of sodium chloride (Nos. 75 and 73, 69 and 70).

(*e*) Decreases with an addition of ammonium chloride (No. 84 to be compared with No. 69, and Nos. 74, 76 and 81 to be compared with No. 75).

(2) Iron deposited from an ordinary oxalate electrolyte is always more or less contaminated with organic matter, while the presence of 5 grms. ammonium chloride, at a current density of 1·0 amp./sq. dm. will prevent contamination even when the electrolysis is continued for 24 hours.

TABLE II.

ELECTRO DEPOSITION OF IRON FROM COMPLEX OXALATE ELECTROLYTES.

Containing 0·1947 grm. iron.

No.	Addition.	Cathodic C.D. amps./sq.dm.	Anodic C.D. amps./sq.dm.	Time Hours.	Deposited Grms.	Character of Residue.
67		1·0	12·5	33	0·1960	A greyish black residue left.
69		2·0	2·0	18	0·1952	As in No. 67
70	5 grms. NaCl	2·0	2·0	18	0·1956	A small residue detected by means of the microscope
73	5 grms. NaCl	1·0	1·0	24	0·1953	Negligible amount of organic matter
74	5 grms. NH_4Cl	1·0	1·0	24	**0·1948**	Visible under the microscope
75		1·0	1·0	25	0·1951	As in No. 67
76	10 grms. NH_4Cl	1·0	1·0	25	**0·1948**	None
77		1·0	12·5	24	0·1958	Rich in organic matter
78	5 grms. NH_4Cl	1·0	1·0	24	**0·1947**	None
79		1·0	12·5	12	0·1953	Smaller than in No. 77
81		1·0	12·5	24	0·1958	As in No. 77
82	5 grms. NH_4Cl	2·0	2·0	12	**0·1948**	Negligible residue
84	5 grms. NH_4Cl	2·0	2·0	24	**0·1950**	Small amount organic matter

In Table III. are given the results of a series of depositions made with extreme care and under strictly analytical conditions:—*Volume of electrolyte*: 180 c.c.; *Ammonium oxalate*: 8 grms.; *Electrodes*: Platinum wire-gauze, standard of analytical work; *Current*: 1·0 amp. (equivalent to 1·0 amp./sq. dm. cathodic C.D.); *Temperature*: 25° to 28° C.

TABLE III.

ELECTRO DEPOSITION OF IRON FROM COMPLEX OXALATE ELECTROLYTES.

Containing 0·2283 ± 0·0001 grm. Iron.

No.	Addition.	Time Hours.	Deposited Grms.	Excess Weight Grms.
87		12	0·2288	0·0005
88		12	0·2286	0·0003
89		12	0·2288	0·0005
90	10 grms. NH_4Cl	12	**0·2284**	0·0001
95		11	0·2288	0·0005
96	5 grms. NH_4Cl	11	**0·2283**	None
91		20	0·2291	0·0008
94		20	0·2296	0·0013
92	10 grms. NH_4Cl	20	**0·2283**	None
85		24	0·2293	0·0010
98		6	0·2280	Deposition not quantitative
97	5 grms. NH_4Cl	6	0·2279	,, ,,

NOTE.—Organic matter was left upon dissolution of all the deposits except those formed in the presence of ammonium chloride.

The possible error introduced in pipetting out the samples from the stock solution was reduced to less than 0·0001 grm. calculated on the iron content. The true iron content was determined by titration with a permanganate solution which had been standardized on the purest sodium oxalate, prepared by the Bureau of Standards. The analyses, performed according to the directions given by F. P. Treadwell for the most accurate work of this kind,[2] gave the following results:—

Twenty-five c.c. of Ferrous Sulphate stock solution consumed: (I.) 41·70 c.c., (II.) 41·75 c.c. permanganate.

The blank titration required: 0·075 c.c. permanganate.*

Normality of permanganate solution: 0·9818 *N*.

Twenty-five c.c. of the stock solution contains: 0·2283±0·0001 grm. iron.

These experiments confirm the previous conclusion that contamination of the deposited iron always occurs except when ammonium chloride is dissolved in the electrolyte.

The idea leading to the application of ammonium chloride as an addition agent to the oxalate electrolyte originated from an experimental study by the writer of the significance of colloidal reactions in the processes responsible for the contamination of iron and nickel deposits.[10] In these experiments, where ammonium oxalate or some other organic material was added to a relatively concentrated solution of an inorganic iron salt, it was found that contamination did not take place so readily when ferrous chloride was substituted for ferrous ammonium sulphate in preparing the electrolyte. That the presence of chlorine ions alone is not responsible for the effect, however, is shown by the fact that an addition of sodium chloride to the oxalate electrolyte tends to increase the quantity of organic matter introduced into the deposit (Table II.). The effect of ammonium chloride is probably one of retardation rather than of prevention, since the experiments (Table II.) show that contamination may take place also in the presence of this agent.†

Preliminary experiments seemed to indicate that the use of ferrous chloride in preparing the electrolyte would be preferable to the sulphate. However, this was not further studied, as the iron generally will be present as, or converted into, its sulphate in the course of an analysis.

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* The use of zinc as a reducing agent will explain this relatively high figure.

† The effect of ammonium chloride on the reactions leading to contamination of the metal cannot be fully explained until the exact nature of these reactions has been disclosed.

In the paper already referred to, the writer has advocated the idea of Lambris that the deposited metal (e.g. iron or nickel) acts as a catalyst of reactions involving decomposition of gaseous products formed by cathodic reduction. The presence of ammonium chloride counteracts the tendency of the ordinary oxalate electrolyte to increase in alkalinity as the electrolysis proceeds, and that the alkalinity is an important factor seems to be proved by the fact that the contamination of iron deposits does not take place until the latter stage of the electrolysis. Thus it may be that the effect of the ammonium chloride is one of preventing the formation of certain organic gas products which are formed by electrolytic reduction in the plain oxalate electrolyte and which are responsible for the contamination of the deposit. Recent experiments by Sontag[11] support this view.

Evidently we are here facing electrochemical phenomena which deserve further investigation.

The time factor is of great importance. The point where quantitative deposition has taken place is rather difficult to ascertain on account of the sensitiveness of the reaction for iron (potassium thiocyanate). It is true that, unlike nickel, iron is not contaminated at the beginning of the electrolysis. However, the writer has never been able to discontinue the electrolysis at a point where complete deposition of absolutely pure iron has taken place (as suggested by Verver, *loc. cit.*). The last traces of iron are deposited with great difficulty and only over a relatively long period of time. Table III. shows that 6 hours do not suffice for quantitative deposition, about 8 hours being the minimum, while 10 to 12 hours seem to be preferable. The application of elevated temperature (*e.g.* 50° C.) reduces the time somewhat. However, the writer has always found that as the temperature rises there is a tendency for iron to be deposited as a brownish film on the upper part of the wall and cover of the beaker. As this substance is insoluble in the electrolyte, a loss in iron results. Consequently, deposition at room temperature is to be preferred.

CONCLUSIONS.

The results of the preceding experiments may be summarised thus:

(1) Though the different variables governing the process of electro-deposition of nickel have been varied within wide limits, it has not been possible to prevent contamination of the precipitated metal with organic matter.

(2) Unlike nickel, iron is not contaminated at the beginning of the deposition. Before the iron is completely removed from the electrolyte, however, organic matter starts to build up in the cathodic deposit.

(3) Ammonium chloride present in the electrolyte will prevent contamination of the deposited iron for a period more than sufficient for quantitative precipitation.

(4) The following method is recommended for the electrolytic determination of iron: 8 grms. of ammonium oxalate and 5 grms. of ammonium chloride are added to the electrolyte, which should contain about 0·25 grm. of iron as sulphate. The resulting solution is diluted to 150–200 c.c. with distilled water.

The electrolysis should be continued for about 12 hours (overnight) with a current of 1 ampere, using standard platinum wire-gauze electrodes.

In order to avoid oxidation the deposit should be washed with alcohol and ether before drying in oven at about 80 to 100° C. Blank experiments proved that no organic contamination was caused by the alcohol or ether.

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Research Laboratory of Applied Chemistry,
Massachusetts Institute of Technology,
Cambridge, 39, Mass., U.S.A.

Discussion.

Dr. B. S. Evans said that in the current density seemed rather high to use with an unstirred solution. He asked what happened to the platinum anode, as in the presence of so much ammonium chloride one would rather expect it to be attacked. He was a little doubtful about the explanation offered of the action of the ammonium chloride, *i.e.* that it reduced alkalinity, as in this case one would have expected it to be as effective for nickel as for iron. He thought it significant that the metal which gave trouble in the matter of carbon contamination was the one which above all was noted for absorbing gases, and suggested that the carbonaceous matter was due to decomposition of gas (*e.g.* acetylene) which had been dissolved in the deposited nickel; the experiments carried out for the paper quoted (Frölich, *T.A.E.S.*, 46, 1924) lent countenance to this view.

Lemon Cheese.

By G. D. ELSDON, B.Sc., F.I.C.

(*Read at the Meeting, April* 1, 1925.)

It will probably be generally admitted that lemon cheese consisted originally of sugar, butter, eggs and lemons. The evidence to prove this is as nearly unanimous as such evidence can be. I have made extensive enquiry into the matter, and have come to this conclusion on the following grounds:—

1. Cookery books, although differing somewhat as to the proportions, are unanimous on the point that the constituents should be sugar, butter, eggs and lemons.

2. Many private and professional cooks of ability and experience have all, without exception, given it as their opinion that the bulk of the material should consist of these four ingredients, although one or two have suggested that a small amount of starch, in place of an equivalent quantity of sugar, is desirable in order to prevent "running" when the material is cooked. It should be added, however, that the old method is to put the lemon cheese in the pastry after it is cooked.

3. *The Opinions of the Manufacturers.*—Those manufacturers whom I have interviewed have unanimously agreed that lemon cheese consisted originally of the four substances already mentioned. One manufacturer in Salford has stated that he has given up the manufacture of lemon cheese as he could not compete with other firms whilst continuing to sell a genuine article, and that he refused to sell any other. The same experience has been related by a well-known manufacturer, and also by another firm in the North of England.

4. Retail shopkeepers in the Borough have been questioned as to their ideas on the subject, and the general opinion expressed by them is well summed up in the words of one of them who stated that she could only sell lemon cheese when it was labelled "Home Made." She further stated that she previously made a "Home Made" lemon cheese herself, for sale, which consisted only of the above four ingredients, and that she sold hundreds of pounds of it. She said that she considered that the product that she was then selling (labelled "Home Made," in spite of the fact that she buys it) was a genuine lemon cheese made as she used to make it, although of course, it was not.

There is, of course, at present no definite standard for lemon cheese, but it is in this respect in no different position from most other foods, as in practically all cases the magistrate before whom a case is heard has to fix his own standard on the evidence submitted to him. The trade in lemon cheese is now on a national scale, and has increased enormously in recent years; it is no longer a luxury, being used in large quantities as a substitute for jam; the trade is sufficiently extensive to require a keen supervision and also to provide an adequate defence in case of legal proceedings being taken.

5. The composition of the best commercial articles. One of these is labelled "This lemon cheese GUARANTEED to be made of Choicest Butter, Fresh Lemons, Fresh Eggs, and Pure Sugar only." This is most certainly suggestive as to the opinion of these makers of what lemon cheese should be. The same idea is indicated by labels on other products. Many samples which are by no means satisfactory are labelled in such a way that the purchaser is liable to be misled into thinking that eggs and butter are the principal ingredients. The fact that only quite small proportions of these substances are present does not affect the argument, as the purport of the labels and advertisements would appear to be a suggestion to the public that the articles are better than they really are.

From a careful consideration of various recipes which have been suggested, it would appear that the article domestically known as lemon cheese has a composition somewhat as follows:—

Sugar	55
Butter	14
Eggs	24
Lemon Juice and rind ..	7
	100

I have been able to see the original recipe of a high class manufacturer which, when worked out to percentages, gives the following composition:—

Sugar	54
Butter	11
Eggs	25
Lemon Juice and rind ..	10
	100

a composition which is quite near that suggested above and which was worked out quite independently.

There is good reason to believe that the lemon cheese originally manufactured on the commercial scale was substantially of this quality, but after such a substance had been in course of manufacture for some time and had been made a commercial success many competitors came into being, but, seeing that they made an inferior article, were able to sell at a lower price, thus being able to undercut the original manufacturers. These latter, in order to keep their trade, were forced to reduce the quality of their article. This process of cheapening, so commenced, has gone on and on during the last thirty years, until, at the present time, there are on the market doubtful concoctions containing no eggs, no butter, and little, if any, other fat, and having 30 to 50 per cent. of added water, the presence of which is masked by the addition of "British gum," and possibly gum tragacanth.

As proof of some of these points the following two recipes may be cited:—

Sugar	152 lbs.	160 lbs.
Tartaric acid	1¾ ,,	2 ,,
Glucose	20 ,,	140 ,,
Starches	36 ,,	—
Margarine	27 ,,	36 ,,
*Gum tragacanth solution ..	7½ gallons	—
Water	12 ,,	160 ,,
Flavouring, etc.	1 lb.	1 ,,
Starch gum	—	30 ,,
Eggs	—	9 ,,
Product	=about 460 lbs., *i.e.* about 45 per cent. added water.	=539 lbs. (less evaporation in each case).

It is quite obvious that such products as these have little in common with the article known to the housewife as lemon cheese. Even if it be admitted that there is no rigid standard for lemon cheese, there must, of necessity, come a time when a product of any nature can no longer claim that title—the question to be decided is in regard to the point when this time arrives.

It is contended by some manufacturers that if such substances are sold at a fair price there is no objection to their being called "Lemon Cheese." This cannot be upheld, firstly because, if correct, the identical argument would hold in the case of margarine and butter and, secondly, because if such a principle is once admitted, there can be no guarantee that identical substances will not be sold at varying prices (*i.e.*, that the inferior article will not be sold at the fair price for the better), as, in many cases, different traders frequently disagree in price for the same article. One of the main points about the Food and Drugs Acts is that description and not price covers the sale; competition is allowed for by the possibility of selling the same article at different prices and not by the selling of different articles at the same price. The general public do not always consider that a lower price means an inferior article (and neither, in fact, does it); they put such variations in price down to keen competition, believing that traders will not be allowed to sell widely varying products under the same description.

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* Prepared by soaking 1 lb. of gum tragacanth in about 3½ gallons of water.

DISCUSSION.

Mr. RENDLE said that the cookery books were far from unanimous in their recipes for lemon cheese. He quoted pronounced variations from those given, and mentioned many proposed ingredients not referred to by the author, such as potatoes, cake crumbs, and almonds. He could produce cakes, made from the author's recipe, on the surface of which sugar had crystallised out. He gave widely divergent dictionary definitions of the terms "lemon cheese," "lemon curd," "cheese cakes," etc.

Mr. BLACK said that the "added water" in the manufacturer's recipe would evaporate on cooking until only the percentage in the author's recipe remained. No ingredient in lemon curd was absolutely required by an existing standard, not even eggs. If glucose, margarine and essential oils had been in use they would have appeared in the old cookery books. Modification should be permissible, provided wholesome ingredients were substituted, to place a cheaper article on the market. Manufacturers would not support fraudulent, misleading labels.

Mr. BOSELEY, referring to the recipes quoted, stated that with only 38 per cent. of sugars the sample would not keep. He asked if preservatives were employed, and, if so, which.

Mr. HINKS said that the last-named manufacturer's recipe was admittedly undesirable. Deterioration in quality could not be prevented without standardisation.

Mr. SALAMON said that a limit should be set to the ingenuity of manufacturers to make water "stand up." The public demand for a particular article need not preclude the exposition of a limit on valueless ingredients.

Mr. HAWKINS stated that the article sold should be what the purchaser thought he was buying, even though comparatively worthless. The presence of salicylic acid in lemon curd indicated bad keeping properties. As long as wholesome ingredients were introduced there need not be too great rigidity in fixing a standard.

Mr. BURFORD submitted that so long as the buyer received a non-injurious article it was a matter for the public rather than the chemist.

Mr. PARTRIDGE said the use of tartaric acid as a cheap substitute for citric acid was indefensible.

Mr. BOLTON traced the natural development of lemon cheese recipes. The danger of standardising food products, he said, was that they would tend to descend to the standard, as in the case of butter. A standard was only necessary when the public was defrauded.

The PRESIDENT thought the question could not be disposed of in a short discussion. The choice of lemon cheese as a typical food product was unfortunate. No distinction was made in the recipe between lemon juice and rind. As long as an article was non-injurious and the public was not defrauded there was no need for a standard. He referred to the analogous case of furniture polish, where beeswax and turpentine had given place to paraffin wax and turpentine substitutes without any loss in effectiveness.

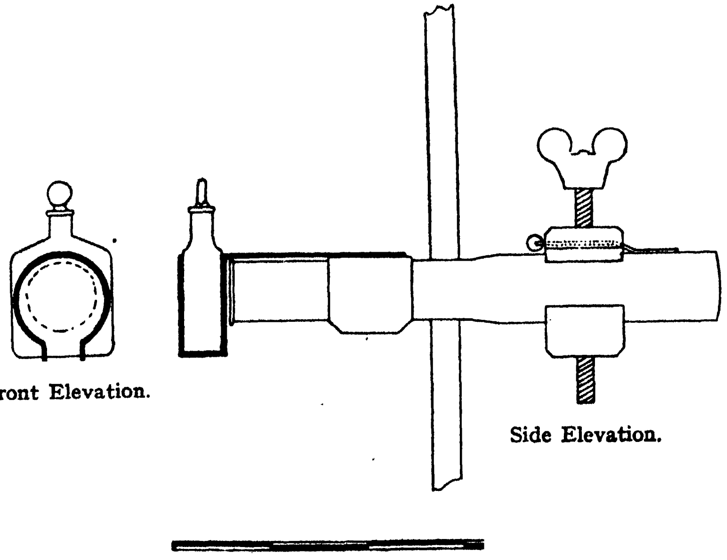
Mr. ELSDON, replying, said that his recipes had been taken from modern cookery books published before, during and since the war. The article supplied to a customer should conform to his demand and with his expectations. Public analysts were presumably appointed with the idea of protecting the public from fraud as well as from injury to health. They should not be merely laboratory workers, but should be at the same time executive and administrative officers, this apparently being the considered opinion of the Society. The preservatives used in the manufacturers' recipes were boric acid and salicylic acid. The moisture content was less evaporation. Synthetic articles ought not to be sold as genuine articles. There was nothing to prevent the manufacturers raising the price of an inferior article. For years he had found very few butters containing excess of moisture. The distinction between lemon juice and rind was not important, as they were used as flavours. He disagreed with the President's views on furniture polish, as obviously if beeswax and turpentine were asked for, beeswax and turpentine should be supplied, whereas if furniture polish were asked for there was, so far as he knew, no generally accepted standard for this substance.

A Simple and Self-Contained Spectroscope Lighting Unit.

By ROBERT C. FREDERICK and E. R. WEBSTER.

(*Shown at the Meeting, March* 4, 1925.)

The apparatus here described is designed for the spectroscopic examination of blood, with particular reference to investigations in suspected cases of carbon monoxide poisoning. In such tests it is essential that the absorption spectrum of the sample, especially during the treatment with the reducing agent, should be made under continuous and simultaneous control by comparison with that of a normal blood under exactly similar conditions. The spectroscope must therefore



Front Elevation.

Side Elevation.

be one with a comparison spectrum, and this, again, calls for duplicate illumination. For this purpose only electric light can be considered satisfactory, but even when available from a main supply its employment presents considerable difficulty. In many instruments the light enters at two orifices, situated immediately at right-angles to each other and neither is more than a centimetre in diameter. The light from two ordinary lamps cannot readily be applied within such a small compass and, moreover, the glare in the observers' eyes interferes with his vision. The blood specimens must be contained in stoppered cells, and in the close proximity of lamps the expansion caused by the heat either blows out the stoppers or, as more frequently happens, fractures the cells.

These drawbacks are avoided by the use of the apparatus shown in the diagram, which is simple, self-contained, efficient, and inexpensive. The light is derived from a small pocket torch with dry battery. The torch is of a type which can be described as simulating the external appearance of a fountain pen of exaggerated diameter; the pocket clip is also the switch. With the clamp covering the clip the torch is firmly secured on a retort stand. The light is switched on and off simply by a fraction of a turn of the clamp screw actuating the switch; this does not alter the rigid hold on the torch. A frame to hold the cell in front of the light is made by bending a piece of brass wire of low flexibility into the shape shown in the smaller sketch; the two free ends are then bent back at right angles a distance slightly less than the thickness of the cell, and then upwards again to give a circular shape such as that made at the commencement. From here the two free ends are again bent back at right angles and taken in contact with each other a distance of about 2 inches. A spring clip is made by bending a piece of tin into the outline of the torch, and on to it the wire frame is soldered. With the frame clipped on to the torch, and the cell held in the wire spring, the apparatus is complete as shown in the larger sketch. One such apparatus provides the direct spectrum, and another, which can be clamped at any necessary angle, the comparison spectrum.

DISCUSSION.

Mr. W. PARTRIDGE mentioned the advisability of choosing a non-smoker as a subject for the spectroscopic test, as carbon monoxide was always present in the blood of a smoker.

Notes.

The Editor wishes to point out that the pages of the Journal are open for the inclusion of short notes dealing with analytical practice and kindred matters. Such notes are submitted to the Publication Committee in the usual manner.

DETERMINATION OF FAT IN CONDENSED MILK.

THE process described by Sutton (ANALYST, 1925, 17) for the determination of fat in condensed milk involves filtration and a cumbersome treatment of filter-paper. In 1907 I published (*Report of the Government Analyst*, Transvaal) a description of a method which avoids these inconveniences, and in which copper-protein, carrying the fat with it, is precipitated as in the process described by Sutton.

For the process 40 grms. of condensed milk (sweetened) are made up with water to 100 ml. Twenty ml. of the mixture are placed in a Leffmann and Beam bottle, and to it are added 5 ml. of Fehling's copper sulphate solution. The mixture is well shaken and centrifuged. The clear solution is separated from the precipitate (which contains all the fat) by means of a narrow-stem pipette. The precipitate is shaken with water, again centrifuged, and the clear liquid is removed. The washing of the precipitate is carried out twice. About 3 ml. of amyl alcohol are run on to the precipitate, and about 10 ml. of water are added. The mixture is then dealt with on the principle of the Gerber process. So much 90 per cent. sulphuric acid is added as will bring the fat layer into the graduated neck of the bottle. The mixture is warmed and centrifugalised: the fat reading is made at about 65° C. When the Leffmann and Beam bottle according to Richmond's specification is used each division (of which there are 80) corresponds with 0·206 per cent. of fat in the condensed milk (the specific gravity of the fat at 65° C. being taken as 0·895).

If it is desired to carry out the determination after the Röse and Gottlieb process, the precipitation, centrifuging and washing may conveniently be carried out in a Stokes, Gottlieb or similar tube, and the precipitate dealt with as described by Sutton (*loc. cit.*).

J. McCRAE.

GOVERNMENT CHEMICAL LABORATORIES,
JOHANNESBURG.

THE SAFEGUARDING OF SEALS.

THE method of sealing samples put forward by Evans (ANALYST, 1925, 130) appears, at first sight, to be simplicity itself, but when carefully examined, it shows defects, which would in all probability have results much more serious than those it is supposed to remedy. The two persons most seriously affected by the method are the inspector and the "referee" analyst. Dealing first with the inspector (presuming that all inspectors are infallible), the method is excellent from his point of view; unfortunately inspectors are not infallible. Firstly he has to carry about with him twelve different bottles of ink and twelve different pens, and he has to remember which pen belongs to each ink, otherwise he will inevitably get some of the wrong secret substance into the other inks; secondly, if he has to mark all three portions of the sample with the same ink, he has twelve different ways of doing it, and he must enter in his book the exact ink used for each sample; if he has to mark each portion of the sample with a different ink, there are something approaching 400 million different ways of doing it, and the possibility of error is enormous in either case.

The provision that the "referee" analyst is to receive the "key" substance privately is also a serious matter. As one who has a considerable number of such samples, taken in many districts, to analyse during the year, I am afraid that this provision is quite unworkable; in many cases the third portion of the sample is not received by the "referee" analyst until a day or two before the summons is to be heard, and it would usually be quite impossible for him to write either to the analyst or to the inspector of the Local Authority concerned and receive an answer and test the seal before the summons is heard. The system of sealing which has been used for many years in the County Borough of Wallasey appears to be very much simpler and less open to the possibility of error than that put forward. In this case the inspector is provided with several seals, each of which bears a secret mark on its face, this mark is not obvious on the seal to a casual examination, but when the seal is carefully examined, as all seals should be, by the "referee" analyst, it is obvious to him, and he is therefore in a position to state definitely that the seal of the sample, examined by him, has not been tampered with and is the same as the seal on the sample, examined by the official analyst; as the inspector takes out with him only one seal, the possibility of error on his part is nil.

This system has stood the test of time, and the possibility of forgery is also reduced to a minimum, as a possible forger would have to have the whole range of seals, as he could never be sure that any particular seal was going to be used; the cost of providing the whole range of seals would be so high that the forger would not be repaid for his trouble.

T. R. Hodgson.

Notes from the Reports of Public Analysts.

The Editor would be glad to receive the Annual or other Reports of Public Analysts containing matter of interest to the Society. Notes made from such Reports would be submitted to the Publication Committee.

HAMMERSMITH BOROUGH COUNCIL.

Annual Report of the Public Analyst for 1924.

Of the 614 samples submitted during the year 1924, 600 were purchased under the Sale of Food and Drugs Acts, and 12 under the Public Health (Milk and Cream Regulations). Of the Food and Drugs samples 508 were reported as genuine, 34 as adulterated, and 58 were of inferior quality.

Milk.—During the year 438 samples were examined, of which 362 were reported as genuine, 58 as inferior, and 18 as adulterated.

Meat and Fish Preparations.—Six of the 20 samples examined contained boron preservative, the amount ranging from 1·8 to 16·1 grains per lb.

Preserved Vegetables.—Seven of 10 samples of peas contained copper salts in quantities ranging from 0·6 grain to 2·5 grains per lb., expressed as crystallised copper sulphate.

Drugs.—Of the 18 drugs examined 2 did not comply with the B.P. requirements. Both of these were samples of borax, one of which contained 200 parts and the other 65 parts of arsenious oxide per million.

P. A. Ellis Richards.

Legal Notes.

Under this heading will be published notes on cases in which points of special legal or chemical interest arise. The Editor would be glad to receive particulars of such cases.

MINERAL MATTER IN CHOCOLATE EGGS.

AN application was made by the Liverpool Corporation to the Liverpool magistrates, on April 3, for the destruction of over 9000 chocolate Easter eggs, which had been found, on analysis, to be unfit for human consumption.

Mr. D. P. Oliver, for the Corporation, said that last July a Liverpool firm had ordered from a firm of cocoa manufacturers in Deptford 23 cwt. of "waste" chocolate in slabs. The greater portion of this had been made into Easter eggs, which had been supplied to various Liverpool retailers. One of these firms had informed an inspector of the Corporation that there was some doubt about some of the eggs, and samples were accordingly submitted to the City Analyst. The analysis showed that they contained 3·5 per cent. of silicious matter, mainly particles of quartz, a few minute fragments of glass, traces of lead, zinc, copper, and a considerable quantity of a woody material of the nature of sawdust. A sample of the original slab chocolate, of which about 3 cwt. remained, was found to contain 3·8 per cent. of silicious matter, mainly quartz, 1·4 grains of lead, and 0·8 grain of zinc per lb., with many minute fragments of metal, some of which were iron and some lead.

The whole of these chocolate eggs had been recalled by their manufacturers, and were in the possession of the Medical Officer.

None of the firms mentioned offered any evidence, and an order for the destruction of the eggs was made.

MISBRANDING OF EMMENAGOGUE PILLS.*

ON February 24, 1924, the United States Attorney for Texas, filed, in the District Court of the United States, a libel praying the seizure and condemnation of 97 boxes of emmenagogue pills which had been shipped from the State of Missouri into the State of Texas, and charging misbranding in violation of the U.S.A. Food and Drugs Acts.

Analyses by the Bureau of Chemistry, U.S. Dept. of Agriculture, of samples of the various articles contained in these packages had given the following results:

La Derma Vagiseptic discs contained salt, alum, starch, milk sugar and talc.

Bick's Nerve Tonic consisted of two products—brown tablets containing phosphorus and compounds of zinc and iron, coated with sugar and calcium carbonate, and yellow pellets containing compounds of iron, strychnine and phosphorus, coated with sugar and calcium carbonate.

Arthur's Sextone Tablets contained iron oxide, calcium carbonate, a compound of zinc, and extract of plant drugs, coated with sugar.

Arthur's Emmenagogue Pills, Leslie's Emmenagogue Pills, and *Thomas's Emmenagogue Pills* contained iron sulphate, aloes, and extract of plant drugs, coated with sugar and calcium carbonate, coloured pink.

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* *U.S. Dept. Agriculture, Service and Regulatory Announcements,* Jan. 31, 1925. Supplement No. 12559.

Bick's Sextone Pills consisted of two products—chocolate-coloured pills containing a small amount of extract of plant drugs, 50 per cent. of sugar, 25 per cent. of calcium carbonate, 7 per cent. of iron oxide, and 7 per cent. of talc; and orange-coloured tablets containing 31 per cent. of metallic iron, 11 per cent. of calcium carbonate, extract of nux vomica and sugar.

Bick's Daisy 99 consisted essentially of extracts of plant drugs, including cascara sagrada and buchu, sodium acetate, alcohol and water.

Bick's Sarsaparilla Compound contained less than 1 per cent. of sodium salicylate, 0·7 per cent. of potassium iodide, extracts of plant drugs, including sarsaparilla and a laxative drug, sugar, alcohol, and water.

Misbranding of the articles was alleged for the reason that the statements appearing in the labelling were false and fraudulent, in that the said products contained no ingredient or combination of ingredients capable of producing the said therapeutic effects.

No claimant having appeared for the property, judgment of condemnation and forfeiture was entered, and it was ordered by the Court that the products be destroyed by the United States Marshal.

Medical Research Council.

INVESTIGATION OF THE SALMONELLA GROUP, WITH SPECIAL REFERENCE TO FOOD POISONING.*

OUTBREAKS of food poisoning and food infection are almost invariably due to infection with a bacillus belonging to the Salmonella group. Practically all the strains investigated fell into one or other of the following types:—Enteritidis type (*B. enteritidis* Gaertner); Derby type; Schottmüller type (*B. paratyphosus* B.); Stanley type; Aertrycke or Mutton type (*B. aertrycke*, probably including the Binnes type strains of Schütze); Newport type; Reading type; Suipestifer or Hog Cholera type (including *B. suipestifer*, *B. paratyphosus* C., *B. Glässer*, and *B. Voldagsen*); Abortus Equi type (*B. abortivo equinus* or *abortus equi*); and *B. paratyphosus* A.

PART I. SEROLOGICAL STUDIES.—To establish the serological identity of two strains, A and B, it is essential to prove:

1. That A supplied in adequate doses is capable of entirely exhausting B serum of agglutinins for B;
2. That B supplied in adequate doses is capable of entirely exhausting A serum of agglutinins for A.

Such proof of identity is essentially qualitative and does not guarantee identical quantitative behaviour. The results of the absorption test are only conclusive when the qualities of the type strains and sera actually employed are known. For this reason the method of working from single colonies was largely used in this research, and the same bacillary agglutination and absorption emulsions were used through each series of experiments. (Details of the technique are given.)

It was observed that agglutination in the Salmonella group was liable to assume two distinct forms—luxuriant flocculation and a fine granulation similar to that seen in the case of *B. dysenteriæ*. The typical Salmonella exhibits two series of antigenic elements: those which are unstable at a temperature much above 60° C. and completely destroyed after heating at 100° C. for 1 to 2 hours, and those which persist unaltered for several hours at 100° C. The former yield with their corresponding agglutinins flocculent agglutination; the latter granulate with the homologous agglutinins.

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* By W. G. Savage, M.D., and P. Bruce White, B.Sc., Pp. 149. Obtainable at Adastral House, Kingsway, W.C.2. Price 3s. 6d. net.

A description is given of the specific and non-specific phases of Salmonella strains and of the stability of these phases, together with an account of cross absorption experiments with these strains and the corresponding sera.

In experiments on the heat-stable Salmonella antigens in toxic canned food, it has been found that repeated injection of animals with saline extracts of the suspected foods will, in certain instances, stimulate the appearance of low-titre agglutinins for Salmonella bacilli. These are almost always purely granulating in action.

As in the case of *B. dysenteriae*, older laboratory cultivations of the Salmonella series may change from their original form of smooth colonies to a granular or "rough" form, and this change appears to be permanent and irreversible. The development of new granulating antigen, peculiar to roughness, tends to be associated with reduction in the quantity and quality of the original antigenic constituents, both flocculating and granulating.

The general conclusion drawn from the seriological work is that the absorption behaviour of the different types affords a practical basis of classification.

PART II. THE CLASSIFICATION AND DISTRIBUTION OF THE SALMONELLA GROUP.—Features common to the types mentioned above are:—They are short sporeless bacilli with rounded ends, possessing flagella and exhibiting motility; gram-negative and staining readily with ordinary dyes; bile tolerance. Culturally they grow as a semi-translucent growth upon agar; grow well in gelatin without liquifying it; grow in milk, with preliminary acid production and subsequent (often delayed) production of marked alkalinity, except in the case of Glässer and Voldagsen types. The milk is never clotted; indol is not produced; lactose, sucrose and salicin are not fermented. In considering inter-classification the most important characters are the biological and serological data, and whilst the serological tests, in particular, have shown that some types are much more nearly related than others, it is not yet possible to adopt an adequately expressive form of grouping. The following summary indicates the distribution and disease-producing rôle of the group:—

B. enteritidis produces in man—gastro-enteritis of food poisoning type; occasionally sporadic cases of illness; in animals, disease of cows, calves and of rats. *Derby type*: in man, food-poisoning; in animals (pigs), exact disease-producing role unknown. *B. paratyphosus* B: in man, paratyphoid fever, probably never food poisoning; in animals, not found. *B. aertrycke*: in man, food poisoning (possibly sporadic cases of illness); in animals, a widespread cause of enteritis in mice, guinea-pigs, and other rodents; occasionally found in pigs; a cause of enteritis in parrots and other birds, and calves; does not occur in rats. *Stanley type*: in man, food poisoning; in animals, not yet isolated. *Newport type*: in man, food poisoning and possibly sporadic cases of illness; in animals: dog suffering from enteritis, otherwise unknown. *B. abortus equi*: in man, not found; in animals, abortion of horses. *B. suipestifer*: in man, paratyphoid fever (C type) and very exceptionally food poisoning; in animals, secondary invader in pigs in hog cholera, and occasionally a cause of disease in pigs.

PART III. STUDIES UPON THE METHODS OF ACTION OF THE SALMONELLA GROUP.—Salmonella food poisoning is, in the majority of cases (probably about three quarters) due to *B. aertrycke.* Next in importance as a causal agent is *B. enteritidis,* possibly causing particularly severe outbreaks, and very likely more prevalent in other countries. The *Newport* type is responsible for a certain number of outbreaks, and the *Stanley* and *Derby* types more rarely. The importance of *B. suipestifer* in this connection is insignificant, and *B. paratyphosus* B. and *B. abortus equi* may probably be altogether discounted. The attack falls mainly on the alimentary tract; general infection is rare, and when it does occur, usually fatal. These food poison bacilli have low invasive powers. In outbreaks apparently associated with dead bacilli (*e.g.* from toxic canned foods) members of the *Salmonella* group are almost certainly implicated. The sera of sufferers seldom show any appreciable development of agglutinins for Salmonella bacilli, and no pathogenic bacilli can be isolated from the food or fæces. D. G. H.

The Presence of Expert Witnesses in Court.

It is customary for expert witnesses, either in civil or criminal cases, to be allowed to remain in court throughout the proceedings, although sometimes they are excluded, like ordinary witnesses. The assumption on which permission for an expert witness to remain is based is that his evidence, usually regarded as a matter of opinion, will not be affected by the evidence of facts given by the other witnesses. This applies more particularly to the chemist than to the medical man, who not infrequently is asked to express an opinion on evidence which he has only just heard.

Referring to this subject in a letter to the *Lancet* (March 28th, p. 685), Sir William Willcox points out that in toxicological cases it is essential for an expert witness to hear the medical evidence, since he may be asked whether the symptoms described are consistent with poisoning by a particular substance. In fact, opposing counsel have, on occasions, observed that they would object to the toxicological evidence if the witness had not heard all the evidence relating to the symptoms. The final decision whether or not any witness shall remain in Court rests with the Judge, but, if the point is raised, previous rulings are taken into consideration.

An outline of some of these rulings, summarised by a legal authority, is given in the letter, from which the following condensed particulars may be cited:

"There is no Statutory regulation on the subject, but it is the usual practice for witnesses who are called to give expert evidence and not to speak to facts to be allowed to remain, even although other witnesses are excluded. If a witness does remain, notwithstanding an order of exclusion, his evidence is not rendered inadmissible, but is the subject of comment.

"The question whether expert witnesses may be allowed to remain in court to hear the evidence is now decided by the kind of questions which may be put to the expert witness, and which assume that he has heard the evidence in the case. In the MacNaghten case (1843) (in which medical men conversant with insanity, who had never seen the prisoner previously, were asked to express an opinion as to his state of mind from the evidence) the Judges to whom the case was referred reported on this point as follows: 'We think the medical man under the circumstances supposed cannot in strictness be asked his opinion in the terms stated, because each of those questions involves the determination of the truth of the facts deposed to which it is for the jury to decide, and the questions are not mere questions upon a matter of science, in which case such evidence is admissible, but where the facts are admitted or not disputed, and the question becomes substantially one of science only, it may be convenient to allow the question to be put in that general form, although the same cannot be insisted on as a matter of right.'

"In the case of *Rex* v. *Wright* (1821) a medical witness was allowed to discuss the facts of the case, and to express an opinion that the prisoner's mind was disordered. This was quoted in the case of *Rex* v. *Searle* (1831), in which a medical witness who had heard all the evidence was allowed to express his opinion as to the sanity of the prisoner.

"In *Russell on Crimes* (8th Edn., p. 2142) there is a note to the effect that: 'It is now the ordinary course to permit not only attorneys, but also professional or scientific persons to remain in Court, the rule being considered as not applying to witnesses of these descriptions.'

"There may well be matters to which neither side attaches importance, which the expert, on hearing them, may deem of great importance. Consequently, I think that, always subject to the discretion of the Judge, but recollecting the established practice, a medical man or other expert should remain in Court, and the value of his assistance in a technical matter to the jury and Court would be much depreciated if he were excluded therefrom."

EDITOR.

ABSTRACTS OF PAPERS PUBLISHED IN OTHER JOURNALS.

Food and Drugs Analysis.

Separation of Egg-yolk into Fractions by Solvents. M. A. Rakusin and G. Pekarskaja. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49**, 39–41.)—A partial separation of the constituents of egg yolk may be effected by extraction with a series of solvents. The yolks are dried in an air oven at 60–70° C., then powdered and first extracted with petroleum spirit, which removes all the egg oil (sp. gr. at 19° C.$=0{\cdot}9403$ $[\alpha]_D=0{\cdot}0$). This is followed by an alcohol extraction, which removes an optically inactive substance containing carbohydrates. The next treatment is with water, after removing the alcohol by warm air; this yields a non-carbohydrate nitrogenous substance giving the biuret reaction and containing tryptophane. The residue is then extracted with 1 per cent. acetic acid, and lastly with 1 per cent. sodium hydroxide solution; the former gives a non-sugar substance showing Adamkiewicz's reaction but not the other protein tests, and the latter extract gives reactions for sugars and the biuret, Millon's and the other protein reactions, except Ostromyslenski's test for amino-bodies. H.E.C.

Determination of Benzoic Acid in Egg-yolk. T. Grethe. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49**, 51–52.)—Ten grms. of the yolk are triturated in a small mortar with 4 c.c. of hydrochloric acid, then 30 grms. of calcined gypsum are ground in, and the mixture dried over sulphuric acid overnight. The dry powder is extracted in a Soxhlet with ether for 2 hours, 2 grms. of dry sodium hydroxide being previously placed in the flask. The ether is distilled off, the residue, which must be alkaline in reaction, is dissolved in warm water, and from this the fat is removed by shaking out with ether in the ordinary way. The alkaline solution is now acidified, and the benzoic acid is extracted with ether, the ethereal solution being washed with water. The ether is distilled at a very low temperature, and the residue of benzoic acid is titrated with standard alkali.

H. E. C.

The Coagulation of Albumin. M. A. Rakusin and A. Rosenfeld. (*Zeitsch. Unters. Nahr. Genussm.*, 1925, **49**, 38–39.)—By heating coagulated egg albumin with five times its weight of water at 101° C. under pressure for 72 hours a partial polymerisation takes place, yielding a solution containing about 1 per cent. of albumin which has an $[\alpha]_D = -39{\cdot}65$. This substance gives all the well known protein reactions of albumin—biuret, xanthoprotein, Millon's, Liebermann's, Adamkiewicz's, Ostromyslenski's, Molisch's, and Pettenkofer's reactions—but it does not coagulate. Ordinary albumin begins to coagulate at 63°. H. E. C.

Detection of Benzoyl Peroxide in Flour and Dough and their Products and in so-called "Improvers." S. Rothenfusser. (*Chem. Zeit.*, 1925, **49**, 285–287.)—The proportion of benzoyl peroxide permitted in flour in Germany, namely 0·005 per cent., is not detectable by the official method, according to which an alcoholic extract of such flour liberates iodine when treated with acidified potassium iodide and starch solution. The detection may be effected, however, by means of a reagent prepared by grinding 1 grm. of *p*-diamino-diphenylamine sulphate in a mortar with 96 per cent. alcohol, rinsing out into a flask, making up to about 100 c.c. with the alcohol, and heating under a reflux condenser for about 30 minutes on a water-bath. On cooling, the alcoholic solution deposits fine crystals, but these are not removed, the liquid being shaken before use. This reagent gives a greenish-blue coloration with benzoyl peroxide.

In the case of a flour, use is conveniently made of a cylinder about 11 cm. in height and 0·8 cm. bore and provided with three marks. The first of these indicates the space occupied by about 0·7 grm. of an average flour when the cylinder is gently tapped on a bench. Petroleum spirit is then added to the second mark, and the cylinder, closed with a cork or the thumb, shaken vigorously for a few seconds, and allowed to drain; more of the petroleum is added to bring the level again to the second mark, about 2·5 c.c. being added in all. After the contents have been gently mixed, from 1 to 1·5 c.c. of the shaken reagent is added (to the third mark) and mixed. If the flour contains 0·01 per cent. of benzoyl peroxide, the petroleum spirit and the alcohol layers are coloured greenish-blue immediately, whilst with smaller proportions the colour may appear only after the lapse of some minutes; with more of the peroxide, the flour itself becomes coloured, any granules of the peroxide being easily visible. If a doughy material is to be tested, a small portion is treated with a few c.c. of petroleum spirit until it crumbles, and 1 to 1·5 c.c. of the reagent then added. T. H. P.

Preparation of Fehling's Solution for the Volumetric Determination of Reducing Sugars. J. H. Lane and L. Eynon. (*J. Soc. Chem. Ind.*, 1925, **44**, 150–152T.)—Seven samples of copper sulphate showed variations in the proportion of contained copper from 98·4 to 99·7 per cent. of that required by the formula, and in the volumetric determination of reducing sugars this is sufficient to be of importance, introducing errors up to 0·7 per cent. of the sugar determined. Elimination of included water from copper sulphate crystals is a tedious operation, and it is preferable to test each stock of copper sulphate by preparing Fehling's solution from it, and titrating this against standard invert sugar solution. When a solution containing 0·5 grm. of invert sugar per 100 c.c., and 25 c.c. of Fehling's solution are used, and with the method previously described (*J. Soc. Chem. Ind.*, 1923, **42**, 32T., and ANALYST, 1923, **48**, 220) 24·8 c.c. of sugar solution should be required, corresponding to a factor of 1240 mgrms., and the adjustment of the copper solution may be made accordingly. D. G. H.

The Fatty Acid Constituents of some Natural Fats. II. Palm Kernel Oil. E. F. Armstrong, J. Allan and C. W. Moore. (*J. Soc. Chem. Ind.*, 1925, **44**, 143–144T.)—Following the procedure detailed in Part I. (*J. Soc. Chem. Ind.*, 1925, **44**, 63T., ANALYST, 1925, 191), the fatty acids prepared from the oil of West African palm kernels were examined and the ethyl esters found to have a saponification equivalent (mol. wt.) of 246·5, and iodine value 14·8. Nine fractions were finally obtained after distillation of the esters under reduced pressure. The "liquid" acids appeared to consist entirely of oleic and linolic acids, and, on esterification, yielded two fractions. The deduced percentage composition of the fatty acids from this palm kernel oil was:—Caprylic acid, 3; capric, 3; lauric, 52; myristic, 15; palmitic, 7·5; stearic (?), 2·5; oleic, 16; and linolic acid, 1.
D. G. H.

The Rancidity of Fats. H. E. Fierz-David. (*Zeitsch. angew. Chem.*, 1925, 6; *J. Soc. Dyers and Col.*, 1925, 115.)—It has been found that ordinary mould fungi, such as *Penicillium glaucum* and *Aspergillus niger*, are capable of hydrolysing fats, the resulting fatty acids being further converted into the same ketones as are obtained by treating them, according to the method of Dakin (*Amer. Chem. J.*, **44**, 41), with hydrogen peroxide and ammonia. The latter process is one of oxidation and takes effect according to the following scheme:—

$$R.CH_2.CH_2.COOH + O \longrightarrow R.CH.OH.CH_2.COOH$$
$$\longrightarrow R.CO.CH_2.COOH \longrightarrow R.CO.CH_3 + CO_2.$$

Thus every fatty acid yielded a characterictic ketone identified by conversion into a semi-carbazone. The rancidity of fats is due, in the absence of bacteria, either to the action of air or to light and moisture, on the unsaturated fatty acids (including possibly also ricinoleic acid), which yield aldehydes and acids, but fats containing saturated fatty acids are oxidised in the presence of moulds to the methylalkyl ketones which are obtainable by Dakin's process. P. H. P.

Detection of Rancidity in Fats and Oils. T. von Fellenberg. (*Mitt. Lebensmittelunters. Hyg.*, 1924, **15**, 198–208; *J. Soc. Chem. Ind.*, 1925, **44**, B179.)—The reagent, which should be stored in the dark and not used for 24 hours after making up, consists of 5 grms. of magenta in 800 c.c. of water, 12 grms. of crystallised sodium sulphite, and 100 c.c. of 0·1 *N* hydrochloric acid, in 1 litre. One c.c. of oil or fat in 1 c.c. of petroleum spirit is shaken with 1 to 2 c.c. of the reagent for half a minute, and if, after 10 minutes, no more colour is present than in a solution of 10 to 20 mgrms. of acetaldehyde per litre, flavour and analytical data must be considered. Coloration of the aqueous layer indicates more advanced rancidity than that of the oily layer only. D. G. H.

Biochemical, Bacteriological, etc.

New Histological Reaction for Proteins. M. Romieu. (*Compt. Rend.*, 1925, **180**, 875–877.)—Although there are many reactions for proteins, there is none suitable for application to histological structures; the new reaction depends upon the red-violet colour produced by syrupy phosphoric acid at about 52° C. It is thought probable that the colour is really due to tryptophane. When applied to a section of tissue this reagent colours protoplasm or epithelium a delicate rose tint, similar to that of eosin. Collagen and gelatin are not coloured, but haemoglobin gives a dull brown. The fragments of tissue should be hardened in alcohol or embedded in collodion, then covered with a drop of syrupy phosphoric acid, and warmed in the paraffin stove for a few minutes. The tissue is thereby rendered transparent as by glycerin and can be examined as usual. The colour is permanent for some hours, then gradually turns brown. H. E. C.

Influence of Pasteurisation on the Digestibility of the Proteins and Mineral Constituents of Milk. E. F. Terroine and H. Spindler. (*Compt. Rend.*, 1925, **180**, 868–870.)—The digestibility of the proteins and mineral constituents of milk before and after pasteurising at different temperatures has been investigated by feeding experiments on young pigs. Milk in the raw state and after pasteurising at 63° C. for 25 minutes, or 95° C. for 1 to 2 minutes, or at 75° C. followed by prompt cooling showed the same condition of digestibility both for protein and ash. It is concluded that pasteurisation has no deleterious effect on milk from the point of view of absorption. H. E. C.

Reproductive Potency of Dry Milk as affected by Oxidation. G. C. Supplee and O. D. Dow. (*J. Biol. Chem.*, 1925, **63**, 103–114.)—The results are recorded by charts of a preliminary investigation wherein it is shown that oxidation changes which may take place under conditions prevailing in milk powder, stored in air over long periods, prevent reproduction. Milk powder containing 12 per cent. of fat, packed and kept in an atmosphere of air in hermetically sealed containers for nearly 2 years, does not permit reproduction in white rats when given in quantities varying from 11·7 to 35 per cent. of the ration, but the same powder stored in deoxygenated air for the same period allowed normal reproduction and the rearing of young when given in quantities varying from 23·3 to 35 per cent. of the ration. Reproduction was possible on lower quantities, but rearing of the young was not successful. An atmosphere of carbon dioxide acted similarly to the atmosphere of air. It cannot be stated whether the negative results thus obtained were due to the carbon dioxide or to an oxygen impurity. Milk powder of the same brand, made under the same conditions at the same time of year as that for the storage experiments but held in an atmosphere of air for 7 months, allowed reproduction and successful rearing of the young when given as 41·7 per cent. of the ration. Reproduction on smaller quantities was possible, but rearing was not successful. Successful reproduction and rearing of the young resulted when the butter fat, as supplied by the Just process dry milk, comprised from 2·8 to 5 per cent. of the ration, providing the milk powder had not been subjected to excessive oxidation after manufacture. P. H. P.

Mineral Deficiencies of Milk as shown by Growth and Fertility of White Rats. A. L. Daniels and M. K. Hutton. (*J. Biol. Chem.*, 1925, **63**, 143–156.)—That rats fed exclusively on cow's milk seldom reproduce, and that only a very small percentage of the young born survive appears to be due to the fact that milk is low in certain inorganic substances necessary for the production of a new generation. Experiments carried out over a number of years are described, and tables and charts show the results. The addition of from 7 to 10 grms. of soya bean powder to 1 litre of milk was effective in correcting this deficiency. Even the ash of the soya bean was effective, and an analysis showed that the ash contained aluminium and silicon. The addition to milk of those ususual mineral substances present in milk in low concentration, *i.e.* manganese, fluorine and aluminium, together with sodium silicate, have resulted in the production of five generations of normal young. These four, used alone or in combinations of two and three, were less effective in correcting the nutritive deficiencies of milk than was the mixture of four salts. The addition of aluminium, manganese, fluorine and silicon to a purified ration made to simulate milk, in the same concentration in which they were used in milk, was without effect on the reproductive processes. It is suggested that failure may have been due to a lack of vitamin X in this mixture, or to too little of the added salts. The addition of iron salts and iodine to milk did not give good results. P. H. P.

Colorimetric Determination of Tryptophane by the Vanillin and Hydrochloric Acid Reaction. Quantitative Separation from Indole and Skatole. I. Kraus. (*J. Biol. Chem.*, 1925, **63**, 157–178.)—The vanillin reaction, specific for tryptophane, is more sensitive with tryptophane itself than any method previously cited. By it, tryptophane has been detected in zein and gelatin which have been considered tryptophane-free. Indole and skatole have been quantitatively separated from tryptophane by means of toluene. Homer's barium hydroxide treatment of tryptophane has been shown by the vanillin reaction to introduce a loss of about 20 per cent.; by direct application of the phenol reagent without toluene extraction a loss of 7 per cent. is introduced. In the barium hydroxide hydrolysis of proteins different decomposition products appear than with tryptophane; the mercury precipitation of tryptophane carries these down. Tyrosine comes down, unless its concentration is low, when it is separated from tryptophane by Folin's method by mercury precipitation in sulphuric acid concentration above 3·5 per cent. Tryptophane, when incubated with the diamino acid fraction and U.S.P. pancreatin or glucosamine with hydrochloric acid and U.S.P. pancreatin, has not been recovered quantitatively. The other simple amino-acids do not prevent recovery of tryptophane. It has not been recovered quantitatively from proteins when hydrolysed by acids, barium hydroxide or U.S.P. pancreatin. Experimental details and results are given. P. H. P.

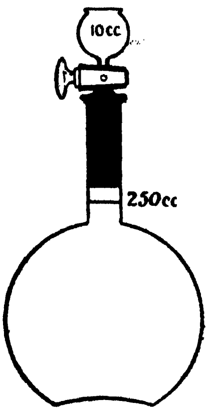
A Protein in the Edible Portion of Orange. A. H. Smith. (*J. Biol. Chem.*, 1925, **63**, 71–73.)—Previous investigations do not give data concerning the nitrogenous constituents, especially the presence and character of protein, in orange juice. The author describes the search for protein in orange juice and some details of its characterisation. In the orange juice as separated from the fruit, containing the orange-red bottom layer, but not the fibrous shreds, there is, at most, 0·2 per cent. of protein, all of which is located in the highly pigmented material contained in the chromatophores of the pulp vesicles. Of the total nitrogen of this material, about 10 per cent. is extracted by strong alcohol and is presumably part of the pigment. Experiments described indicate that the protein found in the pulp of the orange is distinguished from any of the conventional classes of proteins by its unusual solubilities. It is insoluble in water, neutral salt solution, or weak acids, but is soluble in weak alkali. It is not precipitated from its solution by 95 per cent. alcohol, nor is it coagulable by heat at neutral, acid, or alkaline reactions. A polysaccharide, resembling pectin, accompanies the protein fraction throughout the course of the extraction. Since the precipitation-point of this protein is P_H 4·7 and the P_H of orange juice is 4·3, it follows that under ordinary conditions the protein is insoluble within the fruit. Although closely associated with the pigment, the protein is not apparently in chemical combination with it. P. H. P.

Toxicological, Forensic, etc.

Extraction of Alkaloids from Viscera. R. Fabre. (*Compt. Rend.*, 1925, **180**, 966–967.)—The lengthy, though reliable, Stas-Otto extraction may be replaced by proteolysis by pancreatin. The finely divided viscera are pulped with five times their weight of water, boiled for a few minutes, then cooled to 50–55°, and about 2 per cent. of their weight of pancreatin is added and allowed to act at 50–55° for 10–12 hours. The mixture is then boiled, cooled and separated either by filtration or by centrifuging, and the alkaloid or other substance is extracted from the clear solution by shaking out with an immiscible solvent. It is, of course, necessary to be certain that the substance sought for is not affected by pancreatic digestion; strychnine, narcotine, veronal, sulphonal, atropine, cocaine, and morphine are among the substances which can be extracted in this way. H. E. C.

Water Analysis.

Determination of Dissolved Oxygen. J. N. Friend. (*Chem. News*, **1925, 130, 163.**)—The flask shown in the diagram has been devised to overcome certain difficulties in using Winkler's method. The flask is filled to the mark with the water, and 1 c.c. of a solution containing 33 grms. of sodium hydroxide and 10 grms. of potassium iodide per 100 c.c. is added. About 2 c.c. of a manganous chloride solution (40 grms. per 100 c.c.) are now added, sufficient to raise the level of the liquid above the lower end of the stopper and to fill the capillary tube in the stopper when this is pushed home. After the reaction is over in the flask, about 5 c.c. of hydrochloric acid are put into the funnel, the tap opened, and the stopper gradually withdrawn to a sufficient extent to admit the acid without any air. The stopper may now be replaced by a rubber one, and the flask is then shaken and allowed to stand for five minutes, after which the solution is transferred to another flask for the thiosulphate titration. By this means two difficulties are overcome:—(1) Closing the apparatus after introduction of the water and the reacting chemicals without including a bubble of air; (2) opening the apparatus and adding the acid necessary to stop further oxidation, without admitting air. R. F. I.



Action of Water upon Copper Pipes. J. C. Thresh. (*Lancet*, **1925, 208, 675–677.**)—Minute quantities of copper are widely distributed in food and in the human body, and the evidence leads to the conclusion that an amount of copper in water not exceeding 0·1 grain per gallon is not objectionable. The action of water on copper is similar to that on lead, but the solubility of copper oxide is much less than that of lead oxide; filtration removes nearly all the metal from water. The following table shows the action of certain waters on copper pipes determined by the author's well-known method for lead (Analyst, 1921, **46**, 270). The action of hot water is much greater than that of cold water, and the presence of an electric current greatly accelerates the cupro-solvent action. Almost all waters take up copper, but in too small amounts to endanger health.

	Copper parts per 100,000.
River water, moderate temporary hardness.	
London	0·04
Surrey	0·025
Hampshire	0·035
An unfiltered river water	0·06
Deep well waters.	
Chalk	0·000
Limestone	0·05
New red sandstone	0·01
Alkaline water.	
Thanet sand	0·04
Ditto	0·025
Greensand water.	
Hardness 4°	0·01
Moorland water.	
Hardness 4°	0·033 to 0·065
,, 3°	0·01
,, 1°	0·057 to 0·075
,, 1°	0·15
Moorland water (*continued*).	
Hardness 2·75°	0·15
,, 1° (very acid)	0·65
,, acid neutralised with chalk	0·01
2·3°	0·05
4°	0·00
0·5°	0·10
4°	0·045
5°	0·06
Spring waters.	
Hardness 21°	0·05
,, 3°	0·075
,, 15°	0·06
Very pure distilled water.	
Hardness 0°	0·14–0·20
+4 mgrm. $MgCl_2$ Hardness 4°	0·02
+4 mgrm. NaCl Hardness 0°	0·01

H. E. C.

Organic Analysis.

Determination of Carbon in Organic Substances. A. Desgrez and R. Vivario. (*Compt. Rend.*, 1925, **180**, 886–890.)—The wet combustion process has been examined and found to be satisfactory for the determination of carbon in biological fluids, urine, blood plasma, etc. Potassium ferrocyanide and borax efficiently remove all volatile chlorine products, and the oxidation is complete in the presence of the short length of heated copper oxide. The apparatus comprises an aspirator, a 300 c.c. combustion flask fitted with a tap funnel and a reflux condenser from the top of which a tube passes to three U-tubes containing sulphuric acid and pumice, granules of potassium ferrocyanide, and small lumps of fused borax, respectively; then follows a short length of combustion tube containing copper oxide heated to redness, and lastly the usual drying and absorption tubes. The substance to be analysed is placed in the flask with 10 grms. of potassium dichromate, 10 c.c. of water are added, and then, slowly, 125 c.c. of sulphuric acid. The flask is kept gently heated until all evolution of gas ceases, a slow current of air (free from carbon dioxide) being aspirated through the apparatus throughout the experiment. H. E. C.

New Azo-Reaction. F. M. Rowe. (*Chem. Trade J.*, 1925, **10**, 391.)—If a solution of 4-nitrobenzene-2-naphthol-1-diazosulphonate in one molecular proportion of sodium hydroxide be acidified, Para Red can be obtained, but not when an excess of sodium hydroxide is used. In this case, an intense crimson colour is developed which disappears gradually and on subsequent acidification little or no Para Red is obtained, but a compound $C_{16}H_{12}O_7N_3SNa$ can be isolated in a yield of 91½ per cent. Many other diazosulphonates were prepared and tested for this reaction, other diazotisable amino compounds being used in place of *p*-nitraniline. The only compounds not containing a nitro group which gave rise to this new reaction were *p*-aminoazo compounds. R. F. I.

Gravimetric Determination of Picric Acid in a Mixture of Picric Acid and Trinitrometacresol. L. Desvergnes. (*Ann. Chim. anal.*, 1925, **7**, 65–67).—The mixture is dissolved in a warm saturated solution of picric acid in alcohol, and the picric acid which crystallises out on cooling, is weighed. The following identification tests should first be applied:—(1) By warming with a 5 per cent. sodium carbonate solution a clear solution should be obtained. (2) The alkaline solution heated to 80° C. should give a dark red colour. (3) If treated with alkaline hypobromite and bromate and acidified, the mixture should absorb no bromine. The saturated alcoholic solution of picric acid is prepared by heating 100 grms. of pure picric acid in 1 litre of 96 per cent. alcohol till dissolved, and filtering the solution after standing for 3 days at 15° C. The filtrate contains 6·8 to 6·9 grms. of picric acid per 100 c.c., but the exact figure must be determined by evaporation of 50 c.c. *in vacuo.* Twenty-five grms. of the mixture to be examined (powdered and dried *in vacuo*) are heated with 390 c.c. of the picric acid solution (measured at 15° C.) under a reflux condenser till solution is complete, and then left for 5 days at 15° C. The solution is then filtered through a double tared filter on a Buchner funnel, and the crystallised picric acid is dried *in vacuo* and weighed. R. F. I.

Detection and Determination of small Amounts of α-Naphthol in β-Naphthol. T. Callan. (*J. Soc. Chem. Ind.*, 1925, **44**, 125–127.)—An investigation into the sensitiveness of some of the published methods for the detection and determination of α-naphthol in β-naphthol showed that the following 3 tests readily detect as little as 0·2 per cent. of α-naphthol. *Volcy-Boucher test* (*Ann. Chim. anal.*, 1908, **13**, 335).—Half a grm. of the material is dissolved in the smallest possible amount of 45 per cent. alcohol, 2 c.c. of 10 per cent. copper sulphate solution added, and, after shaking, 4 c.c. of a freshly prepared 10 per cent. solution of potassium iodide, when a violet-red precipitate is given by α-naphthol, and a yellow precipitate by β-naphthol. The *Artzberger test* (*Pharm. Post,* **35,** 753; *Pharm. J.*, 1903, **70,** 89).—Three-tenths of a grm. of material are dissolved in 2 to 3 c.c. of alcohol, 10 to 15 c.c. of water added, and, after occasional shaking for 5 to 10 minutes, the solution is filtered, and 10 to 12 drops of 10 per cent. potassium hydroxide solution, 1–4 drops of a solution of 1 grm. of iodine and 2 grms. of potassium iodide in 60 c.c. of water added, when a violet coloration is given by α-naphthol, and a yellow solution by β-naphthol. *The modified B.P. test.*—One tenth of a grm. of the sample is dissolved in 10 c.c. of boiling distilled water, the solution cooled, the crystals of naphthol filtered off, and 0·5 c.c. of a 1 in 30 ferric chloride solution added, drop by drop, to the filtrate, and the colour noted after 15 minutes; *a*-naphthol imparts a violet tinge. The *Liebmann* method (*J. Soc. Chem. Ind.*, 1897, 294) and *Prochazka's* method (*J. Soc. Chem. Ind.*, 1897, 894, and *J. Ind. Eng. Chem.*, 1923, 15, 944), which give quantitative results, depend on the fact that both *a*- and *β*-naphthol couple with diazotized *p*-nitroaniline, to give *p*-nitroazo derivatives soluble in the case of *α*-naphthol in sodium hydroxide solution to a deep blue colour, whilst in the case of the *β*-naphthol the reddish compound is practically insoluble. These methods are capable of determining 0·1 per cent. of *α*-naphthol. Since *α*-naphthol is more soluble in cold water than *β*-naphthol, the sensitiveness of the reaction in tests requiring an aqueous extract is increased. D. G. H.

Determination of Paraffin Wax in Rubber Goods. J. Lagerqirst. (*J. Soc. Chem. Ind.*, 1925, 44, 157–162T.)—The various methods for the determination of paraffin wax are reviewed, and divided into those published previous to 1916, and those published or adopted since that date. Particular attention is paid to the new German method, which is found to give results which are high, particularly when the proportion of paraffin is low, although with softer waxes and with rubber compounds containing vaseline, it is more satisfactory. As a result of experience the following procedure is recommended: The acetone extract from 5 grms. of rubber sample is dissolved in 40 c.c. of absolute alcohol, with the use of a flask with a reflux condenser, and any undissolved sulphur removed by filtering, the flask and filter being washed with 10 to 20 c.c. of boiling absolute alcohol. If, after cooling for 1 hour in a mixture of ice and salt at 4 to 5° C. no flocculent precipitate appears, and there are no indications of mineral oil, the absence of paraffin may be assumed. If present, the precipitate is filtered through a cooled funnel, washed with 50 to 60 c.c. of 90 per cent. (vol.) alcohol, dissolved in warm benzene, and transferred to a weighed flask. The benzene is distilled, and the residue dried and weighed. The filtrate is evaporated to dryness, the residue dissolved in 10 c.c. of carbon tetrachloride, transferred to a separating funnel, and the flask washed out with 10 to 15 c.c. of carbon tetrachloride, followed by 20 to 25 c.c. of concentrated sulphuric acid, both of which are poured into the funnel, and, after vigorous shaking, the acid is allowed to settle and is drawn off. The solution is shaken with further portions of acid until the acid remains colourless, and subsequently water and about 100 c.c. of ether are added. Washing with water is continued until the water is no longer acid, and, after the water has separated from the ether, the ethereal solution is decanted into a weighed flask, the solvent distilled, and the residue dried and weighed. Any sulphur in either residue must be determined (by oxidation with nitric acid and precipitation as barium sulphate) and deducted. D. G. H.

Inorganic Analysis.

Buffer Mixtures Prepared without the Use of Standardised Acid or Base. I. M. Kolthoff. (*J. Biol. Chem.*, 1925, 63, 135–141.)—A new set of buffer mixtures that can be prepared with solutions of weighed crystalline substances is described. The standard substances must obey the condition that they can easily be obtained pure. This is the case with succinic acid, acid potassium phosphate and borax. The mixtures of 0·05 molar succinic acid solution and 0·05 molar borax cover a range of P_H from 3·0 to 5·8; those of 0·1 molar primary potassium phosphate and 0·05 molar borax, from P_H 5·8 to 9·2. For physiological work this range of P_H (from 3·0 to 9·2) will usually suffice. Two graphs, one for succinic acid and borax mixtures and one for KH_2PO_4 and borax mixtures are given, in which the P_H values obtained are plotted as ordinates against the composition of the mixtures as abscissæ. Tables also shew the composition of the mixtures. The buffer action of these mixtures is discussed. P. H. P.

Electrolytic Determination of Cadmium. E. Büttgenbach. (*Zeitsch. anal. Chem.*, **1925, 65,** 452–455.)—The following procedure is recommended in preference to the usual deposition from cyanide solution:—The solution, containing 0·2 grm. of cadmium as sulphate in a bulk of about 150 c.c., is treated with 2 c.c. of strong sulphuric acid, then with 10 to 15 per cent. of sodium hydroxide solution until slightly turbid. After addition of a solution of 6 grms. of potassium hydrogen sulphate the liquid is electrolysed at ordinary temperature at 3·8 volt pressure, the anode disc rotating at 300 R.P.M. The metal is deposited in 40 minutes (hydrogen sulphide test). It is washed as usual without interruption of the current. If more cadmium is present, the addition of the bisulphate and the time of deposition must be increased proportionately. W. R. S.

Determination of Bismuth and its Separation from Lead by Pyrogallol. F. Feigl and H. Ordelt. (*Zeitsch. anal. Chem.*, 1925, **65,** 448–451.)—Solutions of bismuth salts are precipitated quantitatively by pyrogallol if the acidity does not exceed 0·1 *N*. The yellow crystalline precipitate has the composition $BiO_3C_6H_3$ (Bi 62·84 per cent.). The acid solution is neutralised with ammonia until a slight permanent turbidity is produced; this is removed with a drop of dilute nitric acid; a strong solution of pyrogallol is added, and the liquid heated to boiling. The precipitate is collected and washed with dilute nitric acid and water on a Gooch or porous crucible, and dried at 110° C. to constant weight. In the presence of lead, the filtrate from the bismuth precipitate is saturated with hydrogen sulphide, and the lead determined as usual. The results shown are very close. W. R. S.

Separation of Calcium and Magnesium. G. Luff. (*Zeitsch. anal. Chem.*, 1925, **65,** 439–448.)—The separation of calcium as oxalate may be accomplished by a single precipitation from a hot, faintly ammoniacal (1 c.c. excess) solution containing 10 grms. of ammonium chloride in a total volume of 200 c.c., to which ammonium oxalate solution is added, drop by drop, an excess being carefully avoided. The solution, before filtration, is kept one hour on the waterbath.

W. R. S.

Determination of Germanium. E. B. Johnson and L. M. Dennis. (*J. Amer. Chem. Soc.*, **1925, 47,** 790–793.)—The precipitation of germanium as disulphide is complete under the conditions usually employed, but the use of nitric acid to convert the disulphide into dioxide may be replaced advantageously by that of hydrogen peroxide. The oxidation should be effected in a platinum vessel, since high results are obtained when glass is used; the hydrogen peroxide must leave no appreciable residue when evaporated. The disulphide precipitate is dissolved by addition to the filter of several small portions of redistilled ammonia solution, the solution being received in a small weighed platinum dish and oxidised cold by 20 c.c. of perhydrol diluted to 3 per cent. strength. The solution is evaporated to dryness at about 105° C., and the residue moistened with concentrated sulphuric acid and gently heated until the acid and ammonium sulphate are driven off. The residual germanium dioxide is ignited until constant in weight.

T. H. P.

Determination of Chloride, Bromide, or Thiocyanate in Presence of Cyanide. E. Schulek. (*Zeitsch. anal. Chem.*, 1925, **65**, 433–435.)—Formaldehyde in excess reacts quantitatively with cyanide, yielding glycolate and hexamethylenetetramine; the reaction may be utilised in the volumetric determination of chloride, bromide, or thiocyanate in presence of cyanide. From 0·1 to 0·3 grm. of the salt mixture is dissolved in 50 c.c. of water in a flask; 5 c.c. of 20 per cent. formaldehyde solution are added. The flask is well shaken, the solution acidified with 5 c.c. of 30 per cent. nitric acid, and treated with excess of 0·05 *N* silver nitrate solution. The excess is measured by Volhard's method with 0·05 *N* thiocyanate solution, ferric nitrate being the indicator. The process is also valuable for the qualitative detection of the above ions in presence of cyanide.

W. R. S.

Organic Reactions at the Surface of Titania. Adsorption of certain Gases by Titania. F. Bischof and H. Adkins. (*J. Amer. Chem. Soc.*, 1925, **47**, 807–826.)—Sabatier and Mailhe's statement that acetic and propionic acids are reduced by formic acid in presence of titania to the corresponding aldehydes (*Compt. Rend.*, 1912, **154**, 561) is found to be erroneous. Various preparations of titania, made in different ways, differ markedly in their catalytic effects on formic and acetic acids, ethyl acetate, ethyl and *n*-butyl alcohols, and ethyl ether. Only some of these preparations are reproducible as far as their relative catalytic activity is concerned, and the surface conditions determining such activity depend, not only on the size and shape of the radicle attached to the titanium when the compound passes into the solid state, but also on the mechanism of the hydrolysis and dehydration by which the catalysts are obtained. Measurement of the adsorption of carbon dioxide, ethylene, ethane, and hydrogen shows that, although the different samples of titania differ greatly in both absolute and relative catalytic activities, no such differences occur in the adsorption of the gases produced in the reactions catalysed. T. H. P.

Separation of Thorium from Zirconium and the Tervalent Rare Earths. L. Fernandes. (*Gazz. Chim. Ital.*, 1925, **55**, 3–6).—The following method of separation, based on the varying solubilities of the double thallous sulphates, gives satisfactory results. The mixed hydroxides, precipitated by means of ammonia, are washed repeatedly with boiling water and dissolved in cold dilute sulphuric acid. The filtered liquid is treated with excess of thallium sulphate, which precipitates almost the whole of the cerium earths. These are removed by filtration, and the filtrate is heated on a water-bath until more than one-third of the solvent is expelled, the last traces of tervalent earths being thus precipitated. When the filtered liquid is cooled, the first fraction, containing the thorium absolutely pure, separates. The liquid is evaporated again until almost concentrated sulphuric acid is obtained, the various thorium fractions separating being removed. The solution is finally heated to boiling to precipitate the last traces of thorium as the normal sulphate, which is removed by filtration through asbestos or a porous crucible. When the filtrate is cooled, the zirconium compounds separate.

T. H. P.

Separation and Determination of the Alkali Metals by the Use of Perchloric Acid. I. Solubilities of the Perchlorates of the Alkali Metals in Mixed Organic Solvents. G. F. Smith. (*J. Amer. Chem. Soc.*, 1925, 47, 762–769; *cf.* ANALYST, 1923, 48, 627.)—The author has determined the solubility at 25° C. of anhydrous sodium, potassium, rubidium, caesium, and ammonium perchlorates, and of monohydrated sodium and trihydrated lithium perchlorates in mixtures of methyl, ethyl, and *n*-butyl alcohols in various proportions with ethyl acetate. The data given indicate the use of mixed solvents to be of advantage in the separation of the alkali metals by means of their perchlorates. In mixtures of two alcohols, the solubilities of the perchlorates are normal, but in mixtures of ethyl acetate with alcohols the solubilities are abnormally high owing to interaction between the two solvents. The composition of the solvent best fitted to any particular separation may be determined from the numerical results furnished; in general, mixtures of absolute (or 93 per cent.) ethyl alcohol and ethyl acetate give the best results, and mixtures of *n*-butyl alcohol and ethyl acetate exhibit the lowest solubilities for these perchlorates and could probably be used in all proportions. The addition of perchloric acid (for its common -ion effect) to mixed solvents with ethyl acetate as major component is of no value. T. H. P.

Separation and Determination of the Alkali Metals by the Use of Perchloric Acid. II. Precise Determination of the Insoluble Alkali Metal Perchlorates. G. F. Smith and J. F. Ross. (*J. Amer. Chem. Soc.*, 1925, 47, 774–781.)—If dried at 150° C., potassium perchlorate, crystallised from its aqueous solution, may contain about 0·3 per cent. of occluded moisture, which is eliminated only at about 350° C. Of caesium, rubidium, and potassium perchlorates, the first occludes the least, and the last the most, perchloric acid; this occlusion is not dependent on the nature of the organic solvent used. Since strong perchloric acid readily dissolves sodium perchlorate, this salt may occur in appreciable proportion in the crystallised potassium salt. The results described in this and the preceding paper lead to the following procedure for the separation of the alkali perchlorates:—The mixed alkali metal chlorides, free from ammonium chloride, are dissolved in water and treated with two or three times the equivalent weight of pure perchloric acid (not less than 1 c.c. of 60–70 per cent. acid), and the solution thus obtained evaporated to dryness on the hot plate in a 150 c.c. Pyrex beaker, any condensed acid on the walls of the beaker being removed by brushing with the flame. The beaker is then cooled somewhat, and the contents dissolved by addition of a minimum of hot water (2 or 3 c.c. usually suffice), and the liquid again evaporated to dryness on the hot plate. From 10 to 20 c.c. of the mixed organic solvent chosen (see preceding Abstract) are added, and the solution digested near the boiling point for two to three minutes. The liquid is cooled, and the supernatant solution decanted through a platinum-sponge filtering crucible or a Gooch asbestos crucible, and the residue first washed thrice by decantation to remove most of the soluble perchlorate and then dissolved in the minimum of hot water, and the solution again evaporated. The dried salts are extracted, as before, with 10 c.c. of solvent, digested, cooled, and filtered into the original crucible. The precipitate is transferred to the crucible by means of a wash-bottle containing the same solvent, and the contents of the crucible washed with ten to fifteen portions of 0·5–1 c.c. each. The filtrate, which should amount to 35 to 45 c.c., is concentrated on the hot plate preparatory to further analysis. The precipitating beaker is returned to the hot plate until dry, and any remaining particles of perchlorate then brushed into the crucible, which is dried for a few minutes at 110° C. and finally for 15 minutes in a muffle at 350° C. When no sodium or lithium perchlorate is present, the second precipitation and extraction may be omitted. T. H. P.

Separation of Selenium and Tellurium by Sulphur Dioxide in Hydrochloric Acid Solution. V. Lenher and C. H. Kao. (*J. Amer. Chem. Soc.*, 1925, **47**, 769–772.)—In this separation the amount and the concentration of the hydrochloric acid present are of great importance, selenium monochloride not being formed in the cold when concentrated hydrochloric acid is used with a large excess of sulphur dioxide. The procedure recommended is as follows:—A quantity of the mixed oxides of the two elements containing not more than 0·25 grm. of either selenium or tellurium is dissolved in 100 c.c. of cold concentrated hydrochloric acid, and 50 c.c. of concentrated hydrochloric acid saturated at ordinary temperature with sulphur dioxide are then added, with constant stirring. When the red selenium formed subsides, the solution is filtered through an asbestos filter into a filtering flask, and the precipitate washed thoroughly with cold, concentrated hydrochloric acid, then with cold water until all chlorine is removed, afterwards with alcohol, and finally with ether. The selenium is dried for 3 to 4 hours at 30–40° C., and for 1 to 2 hours at 120° to 130° C., and weighed. The filtrate is concentrated to 50 c.c. on a steam-bath below 100° C. to remove excess of acid, and the tellurium afterwards precipitated by the Lenher and Homberger method:—Fifteen c.c. of saturated sulphur dioxide solution are added, and then 10 c.c. of 15 per cent. hydrazine hydrochloride solution, this being followed by 25 c.c. of saturated sulphur dioxide solution. The liquid is boiled, and the precipitated tellurium washed on a Gooch crucible with hot water until all chlorine is removed, and then rapidly with alcohol; the crucible and its contents are dried at 105° C. Under these conditions the tellurium does not oxidise, and the results obtained are more accurate than those given by any other method. T. H. P.

Physical Methods, Apparatus, etc.

The Adsorption of Indicator (Cresol Red) by Serum in the Spectrophotometric Determination of the P_H. E. F. Hirsch. (*J. Biol. Chem.*, 1925, **63**, 55–59.)—In order to determine whether the spectrophotometric method could be used in measuring the P_H of sera or plasmas, cresol red was chosen as indicator, because the usual variations in the reaction of the blood are within the middle and lower portions of the P_H range (6·4 to 9·4) of the indicator. The amount of cresol red necessary for correct transmission values with the spectrophotometric determination of the H-ion concentration of sera varies for different species, and even with sera of individuals from the same species. The amount in rabbit serum is approximately the same as that necessary in the ordinary buffer solutions, but with human serum it is more, and with sheep serum the quantity is considerably greater. A table shews the results. A certain amount of the indicator seems to be bound by some of the serum constituents. When this affinity has been satisfied, or an equilibrium established, there is needed a definite amount unbound which undergoes the tautomeric changes incident to P_H variations. These differences in the quantity of indicator bound by sera may be associated with species specificity.
P. H. P.

Fluorescence as a criterion of purity in organic compounds. E. Bayle and R. Fabre. (*J. Pharm. Chim.*, 1925, **8**, 248–253.)—Fluorescence is measured by the curve of spectral distribution of the intensity of the emissions, by plotting the different radiations of equal intensity as functions of the length of the waves. If the ordinate corresponding to the maximum degree of sensitiveness of the eye for the wave length *a* is taken as 5·550 Angström units, definite bell-shaped curves are obtained. If the intensity of the light of fluorescence is compared with that of white light for each radiation, a curve is obtained, and if the ordinates of this curve are multiplied by the corresponding ordinates for the visibility curve, then the distribution of the intensity of the light in the fluorescent spectrum is defined. This may be of importance in ascertaining the purity of many crystalline bodies, and the method has been applied, in particular, to hydrastine which is very fluorescent, but of which the derivatives, with the exception of opianic acid, are non-fluorescent. Incidentally, the strength of unstable solutions may also thus be ascertained (*cf.* ANALYST, 1924, **49**, 454). D. G. H.

Reviews.

NEW REDUCTION METHODS IN VOLUMETRIC ANALYSIS. E. KNECHT and E. HIBBERT. Pp. x + 134. London: Longmans, Green & Co. Price 8s. 6d. net.

The second edition of Knecht and Hibbert's book is very welcome. The extraordinary reducing power of titanous chloride has made it very desirable that its numerous applications to analytical processes should be collected together in an easily accessible form. This the authors have succeeded in doing.

The subject matter has been rearranged, and every endeavour has been made to bring it up to date by including work from foreign papers and introducing a number of new estimations, among which may be mentioned those of vanadium and phenylhydrazine. The range of determinations covered is very large, as can be judged by the following selection: Copper, chromium (separately and together), perchlorates, nitro compounds, various dye stuffs and sugars. A glance at the index shows what a large number of determinations, otherwise doubtful or impossible, can be made by means of titanous chloride, and in the text one finds actual figures obtained from analyses of samples of known purity, and hence it is easy to judge the degree of accuracy to be expected. Finally, attention may be drawn to the excellent list of original papers and to the subject and author indexes.

The book is a very useful addition to the literature of analytical chemistry, and should prove of great value. HAROLD TOMS.

CHARTS OF THE CHEMICAL REACTIONS OF THE COMMON ELEMENTS. By JOHN A. TIMM, Ph.D. Pp. ix.+81. New York: John Wiley & Sons, Inc.; London: Chapman & Hall. Price 10s. net.

In this book are arranged, in alphabetical order, charts indicating the main reactions of the common elements. Each chart is followed by a list of these reactions expressed in equation form. The object of this volume, so we read in the preface, is to enable a teacher to set his students periodic and rapid reviews of his course. Possibly the book is useful for this purpose and it may be that students working for the London Matriculation will find these charts and lists of equations helpful. HAROLD TOMS.

BITUMINOUS SUBSTANCES. By P. E. SPIELMAN, Ph.D., F.I.C. Pp. xv.+206. London: E. Benn, Ltd. 1925. Price 15s. net.

In writing this book Dr. Spielman has set himself the task of bringing together in one volume an account of the chemical and physical investigations carried out on bitumens during the last 15 years. The use of bitumen as asphalt in roadmaking has increased enormously during this period but, though the amount of scientific work has not been small, the problem of the composition of the different known varieties is still far from being solved. Dr. Spielman's book, therefore, in giving a full account of the present position of scientific knowledge, serves as a milestone in the progress of the chemistry of bituminous materials.

The book commences with an interesting chapter on the first references in early history to the use of bitumen. The second and third chapters review the present knowledge of the origin of the different varieties of bitumen and of their composition and give an account of their physical and chemical properties. The effect of heat on constitution and properties is discussed at length, but so far the extent of the changes which are described appears not to have been determined, except in a very few instances.

The physical and chemical tests on bitumens are fully described in Chapter V., and should be read in conjunction with the Appendix giving the standard tests devised by the Institution of Petroleum Technologists, 1924. Many of the tests are empirical ones, and the correlation of the results with one another is carefully described and discussed.

The Appendix, apart from the above standard tests, consists of a discussion of the nomenclature which has been applied to bituminous substances by the various workers in the field. This chapter is particularly interesting in that it illuminates the views of the various authorities. In this respect it should be read carefully along with the view expressed in the chapter on composition and origin.

The book is well written, and the bibliography is full and well arranged. The subject-matter should prove of great interest to all enquirers on this subject, and be of considerable assistance to all scientific workers in this field.

The publishers are to be complimented on having produced a compact volume, well printed in very clear type, at a reasonable cost. J. G. KING.

SYNTHETIC ORGANIC COMPOUNDS. By S. P. SCHOTZ, D.Sc. Pp. 412. London: Ernest Benn, Ltd. 1925. Price 45s. net.

This is a very interesting book and it should prove of much value to the average student of organic chemistry. It will give him an idea how organic substances are prepared in industry, and it will also make him realise the differences between laboratory and factory work. The book is obviously not written for the industrial chemist, since it is quite impossible for one author to deal with such a variety of subjects as perfumes, synthetic tannins, artificial silk, explosives, etc. Industrial chemists will therefore be disappointed, as was the reviewer, when he read the article on synthetic tannins. Incidentally, it might be mentioned that it was not Stiasny (1912), but Baeyer, who discovered, in 1872, that formaldehyde condenses with phenols to give a product which precipitates gelatin, and it was this discovery which led Stiasny to patent his syntans.

The book is well, if not luxuriously, printed. Much cost could have been saved, however, if more space had been economised in the printing of the book. One wonders if the graphical formulæ for phenyl salicylate (p. 134), phenyl carbonate (p. 136), phthalic anhydride (p. 209), Baekland's formulæ for resin (pp. 383, 384) are really required? The omission of such extravagances would surely have reduced the cost of the book. After all, 45s. is a high price to be paid for a book which can·only be of passing interest. M. NIERENSTEIN.

COLLOID CHEMISTRY. By JEROME ALEXANDER, M.Sc. 2nd Edition. Pp. 208+viii. London: Chapman & Hall. 1925. Price 9s. 6d. net.

Mr. Jerome Alexander's treatise on Colloid Chemistry serves two useful purposes. In the first place it puts forth in a clear and interesting manner for the general reader how the work of the chemist in his laboratory forms the foundations of the great industries of civilisation; and in the second place it points out to the chemist himself how the results of laboratory experiments can be applied and adapted in wider and wider spheres. The full title of Mr. Alexander's book, set out on the title page, is "Colloid Chemistry, an Introduction and some Applications." The abbreviated title on the cover suggests a text book merely, and is a little misleading, for although this new edition may serve as a text book, there is no doubt that its chief merit, as in the former edition, lies in the masterly review of the manifold applications of colloid chemistry to civilisation. Mr. Alexander has a judicial mind. He asks, "What is colloid chemistry?" and then he picks out the salient points from the mass of evidence before him, and, while ignoring nothing of importance, answers the question in fifty-four pages. The second chapter, in which he makes sure that his readers get an understanding of the mental tools which they will be called upon to use, is especially to be commended. These tools, as in every other branch of exact science, are the special methods of analysis and measurement that are required by the nature of the subject. Without an understanding of them, progress on the theoretical side is impossible.

In any matter of colloidal theory "where doctors disagree," all sides are given a fair hearing, and though there is never any doubt as to Mr. Alexander's intellectual preference, he never descends to being partisan.

The remaining 140 pages are devoted to the applications of colloid chemistry, and the subjects range over medicine, physiology, food, clothing, dyes, explosives, coal and steel.

Mr. Alexander is editing a monograph on the theory and practice of colloid chemistry. The tantalising glimpses at the subject in this little volume provoke an eager longing for the appearance of the big one. D. JORDAN LLOYD.

CHEMISTRY IN THE SERVICE OF MAN. By ALEXANDER FINDLAY, M.A., D.Sc., F.I.C. 3rd Edition. Pp. 300. London: Longmans, Green & Co. Price 6s. net.

During recent years there has been a considerable output, by men of science, of books in which the bearing of science on everyday life has been set forth in such a manner that it can be appreciated by the intelligent lay reader. The fact that such books pass into second and even later editions is evidence that the public is quite willing to take an interest in science, provided its ideas come to it expressed in a language it can understand.

Professor Alexander Findlay's *Chemistry in the Service of Man* has now deservedly reached a third edition. Three new chapters have been added on "Radioactivity and Atomic Structure," "The Rare Gases of the Atmosphere," and "The Metals and their Alloys." An understanding of modern theories of radioactivity and atomic structure has now become a necessity for the chemist, and the story of the discovery of the isotopes of the elements is an interesting instance of how an apparent difficulty in harmonising the theoretical requirements of integral atomic weights with the fractional values obtained by accurate analysis led to one of the greatest advances in knowledge, since the original discovery of the chemical equivalence of the elements. The story of the discovery of the rare gases of the atmosphere, again, is an instance of the value of the correct interpretation of accurate analytical results in leading to new knowledge and discoveries. The chapter on the metals will be enlightening to many people now that the expression "non-ferrous metals" has become a political catch-word.

Of the book, as a whole, it may be said that it gives an able exposition of its subject. Professor Findlay's style is particularly easy to read. The unlaboured presentment which he achieves is only possible where the writer has a firm grip on his subject.

It is a pity that the publishers have found it necessary in this new edition to alter the type. The combination of smaller fount and coarser paper, while leaving the bulk of the volume almost unaltered, imposes a considerably increased strain on the eye.

D. JORDAN LLOYD.

A GERMAN-ENGLISH DICTIONARY FOR CHEMISTS. By AUSTIN M. PATTERSON, Ph.D. Pp. 343. New York: John Wiley & Sons; London: Chapman & Hall. 1924. Price 12s. 6d. net.

The first edition of this excellent little dictionary was published in 1917, and it has now been reprinted for the fifth time, bringing the total issue up to twenty-one thousand. The present issue, however, is not merely a reprint of the first, for it contains 26 pages of addenda, also arranged alphabetically, giving a large number of less common words or of those used in a specialised sense. This addition adds greatly to the value of the book. Not only are purely chemical terms given, in this or the older portion, but there are also numerous technical words used in anatomy, botany, medicine and various crafts, such as ceramics, dyeing and printing, and a useful feature, not to be found in the large general dictionaries, is the inclusion of the various abbreviations used in German chemical and technical journals.

In warmly recommending this dictionary to chemists the reviewer can speak from experience, for he has had the first issue in constant use for some years, and has seldom found it wanting, even when the word sought has been an obsolete or unusual one.

EDITOR.

JUNE, 1925. Vol. L., No. 591.

THE ANALYST.

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PROCEEDINGS OF THE SOCIETY OF PUBLIC ANALYSTS AND OTHER ANALYTICAL CHEMISTS.

An Ordinary Meeting of the Society was held at the Chemical Society's Rooms, Burlington House, on Wednesday, 6th May, 1925. The President, Mr. G. Rudd Thompson, F.I.C., was in the chair.

A certificate was read for the first time in favour of Mr. Theodore Rendle.

Certificates were read for the second time in favour of:—Messrs. Lewis Eynon, B.Sc., F.I.C., Jack Rowan Heather, Frederick George Hitchman, and William David Rogers, B.Sc., A.R.C.S., F.I.C.

The following were elected members of the Society:—Messrs. George William Fraser Holroyd, M.A. (Oxon.), F.I.C., Cecil Eric Keeley, Andrew Francis Macculloch M.A., B.Sc. (Edin.), A.I.C., Frank Vegetus North Mitchell, Charles Henry Thomson, and Walter Peter Whitley, B.A. (Oxon).

The following papers were read:—"The Adulteration of Conserves, with Special Reference to Pectin and Agar-Agar," by John King, F.I.C.; "The Influence of Palm Kernel Meal on the Composition of Bacon Fat," by J. S. Willcox and H. T. Cranfield; "Points Arising from the Analytical Standardisation of British Chemical Standards," by C. H. Ridsdale, F.I.C., and N. D. Ridsdale; "A New Method for the Separation and Determination of Tin in Alloys," and "A New Colorimetric Method for the Determination of Cobalt in the Presence of Nickel," by B. S. Evans, M.C., M.B.E., Ph.D., B.Sc., F.I.C.; and "The Determination of Small Amounts of Iron by Colorimetric Methods," by W. B. Walker, B.Sc., A.I.C.

The Determination of Oxides of Nitrogen (except Nitrous Oxide) in Small Concentration in the Products of Combustion of Coal Gas and in Air.

By A. G. FRANCIS, B.Sc., F.I.C., AND A. T. PARSONS, B.Sc., A.I.C.

INTRODUCTION.—For the purpose of an investigation a method was needed to determine oxides of nitrogen both in the products of combustion of coal gas and in these products when largely diluted with air. The method, therefore, should combine delicacy with flexibility, since the proportions of oxides of nitrogen to be determined might vary from less than 1 part in a million to as much as 100 or 200 parts in 1,000,000. Methods depending on the precipitation of nitron nitrate and on the reduction of nitric acid to ammonia were clearly inadmissible unless very large quantities of the products of combustion could be worked up.

The process of Allison, Parker and Jones (*Technical Paper* 249, Bureau of Mines, Washington), was examined, as it appeared to be suitable for the purpose. This depends on absorbing the oxides of nitrogen in a closed bottle by 5 ml. of a 10 per cent. sodium hydroxide solution and 5 ml. of hydrogen peroxide (no strength stated) during 30 minutes, the resulting liquid being subsequently evaporated to dryness in a beaker over an electric hot plate. The dry solid is moistened with 2 ml. of a 50 per cent. solution of phenol-disulphonic acid in concentrated sulphuric acid, diluted to 10 ml. with distilled water, filtered, made ammoniacal with 15 ml. of concentrated ammonia solution diluted with water (1:1), and its volume made up to 100 ml. The colour is then compared in Nessler tubes with that developed by standard solutions of potassium nitrate. A blank determination on the reagents is made at the same time.

In the paper quoted no mention is made of the process having been tested upon mixtures of oxides of nitrogen and air. On making this comparison, results far below the truth were obtained, and it was also found that the alkaline solution of the absorbed oxides of nitrogen, after having been oxidised by hydrogen peroxide in several concentrations, still contained large quantities of nitrite when tested with the Griess-Ilosvay solution. Attention was consequently directed to:—(A) The absorption of oxides of nitrogen by hydrogen peroxide and by caustic alkali: (*a*) in a closed bottle; (*b*) by scrubbing. (B) The oxidation of sodium nitrite by means of hydrogen peroxide.

A. THE ABSORPTION OF OXIDES OF NITROGEN BY HYDROGEN PEROXIDE AND BY CAUSTIC ALKALI.

As it seemed from the preliminary experiments that nitric oxide in air escaped complete oxidation in alkaline hydrogen peroxide, further experiments were made to test the absorption of oxides of nitrogen by hydrogen peroxide and by

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